

SUBCHAPTER D—WATER PROGRAMS (CONTINUED)

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

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APPENDIX D TO PART 136—PRECISION AND RECOVERY STATEMENTS FOR METHODS FOR MEASURING METALS

AUTHORITY: Secs. 301, 304(h), 307 and 501(a), Pub. L. 95-217, 91 Stat. 1566, *et seq.* (33 U.S.C. 1251, *et seq.*) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

§ 136.1 Applicability.

(a) The procedures prescribed herein shall, except as noted in §§ 136.4, 136.5, and 136.6, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(1) An application submitted to the Administrator, or to a State having an approved NPDES program for a permit under section 402 of the Clean Water Act of 1977, as amended (CWA), and/or to reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under parts 122 to 125 of title 40; and

(2) Reports required to be submitted by dischargers under the NPDES established by parts 124 and 125 of this chapter; and

(3) Certifications issued by States pursuant to section 401 of the CWA, as amended.

(b) The procedure prescribed herein and in part 503 of title 40 shall be used to perform the measurements required for an application submitted to the Administrator or to a State for a sewage sludge permit under section 405(f) of the Clean Water Act and for record-keeping and reporting requirements under part 503 of title 40.

[72 FR 14224, Mar. 26, 2007, as amended at 77 FR 29771, May 18, 2012]

§ 136.2 Definitions.

As used in this part, the term:

(a) *Act* means the Clean Water Act of 1977, Pub. L. 95-217, 91 Stat. 1566, *et seq.* (33 U.S.C. 1251 *et seq.*) (The Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

(b) *Administrator* means the Administrator of the U.S. Environmental Protection Agency.

(c) *Regional Administrator* means one of the EPA Regional Administrators.

(d) *Director* means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) *National Pollutant Discharge Elimination System (NPDES)* means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) *Detection limit* means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth at appendix B of this part.

[38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 26, 1984]

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed

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together with test procedure descriptions and references in Tables IA, IB, IC, ID, IE, IF, IG, and IH. The methods listed in Tables IA, IB, IC, ID, IE, IF, IG, and IH are incorporated by reference, see paragraph (b) of this section, with the exception of EPA Methods 200.7, 601–613, 624, 625, 1613, 1624, and 1625. The full texts of Methods 601–613, 624, 625, 1613, 1624, and 1625 are printed in appendix A of this part 136, and the full text of Method 200.7 is printed in appendix C of this part 136. The full text for determining the method detection limit when using the test procedures is given in appendix B of this part 136. The full text of Method 200.7 is printed in appendix C of this part 136. In the event of a conflict between the reporting requirements of 40 CFR parts 122 and 125 and any reporting requirements associated with the methods

listed in these tables, the provisions of 40 CFR parts 122 and 125 are controlling and will determine a permittee's reporting requirements. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, IF, IG, and IH. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, ID, IE, IF, IG, and IH or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and §§ 136.4 and 136.5. Under certain circumstances paragraph (c) of this section, § 136.5(a) through (d) or 40 CFR 401.13, other additional or alternate test procedures may be used.

TABLE IA—LIST OF APPROVED BIOLOGICAL METHODS FOR WASTEWATER AND SEWAGE SLUDGE

Parameter and units	Method ¹	EPA	Standard methods	AOAC, ASTM, USGS	Other
Bacteria:					
1. Coliform (fecal), number per 100 mL or number per gram dry weight.	Most Probable Number (MPN), 5 tube, 3 dilution, or Membrane filter (MF) ² , single step	p. 132 ³ 1680 ^{11,15} , 1681 ^{11,20} , p. 124 ³	9221 C E–2006. 9222 D–1997 ...	B–0050–85 ⁴	
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 132 ³	9221 C E–2006.		
3. Coliform (total), number per 100 mL.	MF ² , single step ⁵ MPN, 5 tube, 3 dilution, or.	p. 124 ³ p. 114 ³	9222 D–1997. 9221 B–2006.	B–0025–85 ⁴	
4. Coliform (total), in presence of chlorine, number per 100 mL.	MF ² , single step or two step. MPN, 5 tube, 3 dilution, or	p. 108 ³ p. 114 ³	9222 B–1997 ... 9221 B–2006		
5. <i>E. coli</i> , number per 100 mL ²¹	MF ² with enrichment ⁵ MPN ^{6,8,16} multiple tube, or.	p. 111 ³	9222 (B + B.5c)–1997. 9221B.1–2006/ 9221F–2006 ^{12,14} ,		
	multiple tube/multiple well, or	9223 B–2004 ¹³	991.15 ¹⁰ ...	Colilert® ^{13,18} Colilert–18 ^{®13,17,18}
	MF ^{2,6,7,8} single step ...	1603 ²²	mColiBlue–24 ^{®19}
6. Fecal streptococci, number per 100 mL.	MPN, 5 tube 3 dilution, or MF ² , or	p. 139 ³ p. 136 ³	9230 B–2007. 9230 C–2007 ...	B–0055–85 ⁴	
7. Enterococci, number per 100 mL ²² .	Plate count MPN ^{6,8} , multiple tube/multiple well, or MF ^{2,6,7,8} single step or	p. 143 ³	D6503–99 ⁹	Enterolert® ^{13,24}
8. <i>Salmonella</i> , number per gram dry weight ¹¹ .	Plate count MPN multiple tube	1600 ²⁵ p. 143 ³ . 1682 ²³ .	9230 C–2007		
Aquatic Toxicity:					

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TABLE IA—LIST OF APPROVED BIOLOGICAL METHODS FOR WASTEWATER AND SEWAGE SLUDGE—Continued

Parameter and units	Method ¹	EPA	Standard meth-ods	AOAC, ASTM, USGS	Other
9. Toxicity, acute, fresh water organisms, LC ₅₀ , percent effluent.	<i>Ceriodaphnia dubia</i> acute. <i>Daphnia pulex</i> and <i>Daphnia magna</i> acute. Fathead Minnow, <i>Pimephales promelas</i> , and Bannerfin shiner, <i>Cyprinella leedsi</i> , acute. Rainbow Trout, <i>Oncorhynchus mykiss</i> , and brook trout, <i>Salvelinus fontinalis</i> , acute.	2002.0. ²⁶ 2021.0. ²⁶ 2000.0. ²⁶ 2019.0. ²⁶			
10. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC ₅₀ , percent effluent.	Mysid, <i>Mysidopsis bahia</i> , acute. Sheepshead Minnow, <i>Cyprinodon variegatus</i> , acute. Silverside, <i>Menidia beryllina</i> , <i>Menidia menidia</i> , and <i>Menidia peninsulae</i> , acute.	2007.0. ²⁶ 2004.0. ²⁶ 2006.0. ²⁶			
11. Toxicity, chronic, fresh water organisms, NOEC or IC ₂₅ , percent effluent.	Fathead minnow, <i>Pimephales promelas</i> , larval survival and growth. Fathead minnow, <i>Pimephales promelas</i> , embryolarval survival and teratogenicity. Daphnia, <i>Ceriodaphnia dubia</i> , survival and reproduction. Green alga, <i>Selenastrum capricornutum</i> , growth.	1000.0. ²⁷ 1001.0. ²⁷ 1002.0. ²⁷ 1003.0. ²⁷			
12. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC ₂₅ , percent effluent.	Sheepshead minnow, <i>Cyprinodon variegatus</i> , larval survival and growth. Sheepshead minnow, <i>Cyprinodon variegatus</i> , embryolarval survival and teratogenicity. Inland silverside, <i>Menidia beryllina</i> , larval survival and growth. Mysid, <i>Mysidopsis bahia</i> , survival, growth, and fecundity.	1004.0. ²⁸ 1005.0. ²⁸ 1006.0. ²⁸ 1007.0. ²⁸			

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TABLE IA—LIST OF APPROVED BIOLOGICAL METHODS FOR WASTEWATER AND SEWAGE SLUDGE—Continued

Parameter and units	Method ¹	EPA	Standard methods	AOAC, ASTM, USGS	Other
Sea urchin, <i>Arbacia punctulata</i> , fertilization.	1008.0. ²⁸				

Table IA notes:

- ¹ The method must be specified when results are reported.
- ² A 0.45-µm membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.
- ³ Microbiological Methods for Monitoring the Environment, Water, and Wastes, EPA/600/8-78/017. 1978. US EPA.
- ⁴ U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. USGS.
- ⁵ Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.
- ⁶ Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.
- ⁷ When the MF method has been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.
- ⁸ To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.
- ⁹ Annual Book of ASTM Standards—Water and Environmental Technology, Section 11.02. 2000, 1999, 1996. ASTM International.
- ¹⁰ Official Methods of Analysis of AOAC International. 16th Edition, 4th Revision, 1998. AOAC International.
- ¹¹ Recommended for enumeration of target organism in sewage sludge.
- ¹² The multiple-tube fermentation test is used in 9221B.1–2006. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.
- ¹³ These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β-glucuronidase produced by *E. coli*.
- ¹⁴ After prior enrichment in a presumptive medium for total coliform using 9221B.1–2006, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F–2006. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.
- ¹⁵ Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium, EPA–821–R–10–003. April 2010. U.S. EPA.
- ¹⁶ Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple-well procedures, Quanti-Tray®, Quanti-Tray®/2000, and the MPN calculated from the table provided by the manufacturer.
- ¹⁷ Colilert-18® is an optimized formulation of the Colilert® for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 °C rather than the 24 h required for the Colilert® test and is recommended for marine water samples.
- ¹⁸ Descriptions of the Colilert®, Colilert-18®, Quanti-Tray®, and Quanti-Tray®/2000 may be obtained from IDEXX Laboratories, Inc.
- ¹⁹ A description of the mColiBlue24® test, is available from Hach Company.
- ²⁰ Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A–1 Medium, EPA–821–R–06–013. July 2006. U.S. EPA.
- ²¹ Recommended for enumeration of target organism in wastewater effluent.
- ²² Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC), EPA–821–R–09–007. December 2009. U.S. EPA.
- ²³ Method 1682: *Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium, EPA–821–R–06–014. July 2006. U.S. EPA.
- ²⁴ A description of the Enterolert® test may be obtained from IDEXX Laboratories Inc.
- ²⁵ Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), EPA–821–R–09–016. December 2009. U.S. EPA.
- ²⁶ Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. EPA–821–R–02–012. Fifth Edition, October 2002. U.S. EPA.
- ²⁷ Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. EPA–821–R–02–013. Fourth Edition, October 2002. U.S. EPA.
- ²⁸ Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. EPA–821–R–02–014. Third Edition, October 2002. U.S. EPA.

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/Other
1. Acidity, as CaCO ₃ , mg/L.	Electrometric endpoint or phenolphthalein endpoint.....	2310 B–1997.	D1067–06 ..	I–1020–85. ²	
2. Alkalinity, as CaCO ₃ , mg/L.	Electrometric or Colorimetric titration to pH 4.5, Manual.....	2320 B–1997.	D1067–06 ..	973.43 ³ , I–1030–85. ²	
3. Aluminum—Total, ⁴ mg/L.	Automatic Digestion, ⁴ followed by any of the following:	310.2 (Rev. 1974) ¹	I–2030–85. ²

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/ Other
4. Ammonia (as N), mg/L.	AA direct aspiration ³⁶	3111 D–1999 or 3111 E–1999.	I–3051–85. ²
	AA furnace	3113 B–2004.		
	STGFAA	200.9, Rev. 2.2 (1994).	3120 B–1999.	D1976–07 ..	I–4471–97. ⁵⁰
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3125 B–2009.	D5673–05 ..	993.14, ³ I–4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3500–AI B–2001.	D4190–08 ..	See footnote. ³⁴
	Direct Current Plasma (DCP) ³⁶	4500–NH ₃ B–1997.		
	Colorimetric (Eriochrome cyanine R).	4500–NH ₃ F–1997.		
	Manual distillation ⁶ or gas diffusion (pH > 11), followed by any of the following:	350.1, Rev. 2.0 (1993).	4500–NH ₃ C–1997.	973.49 ³ .
	Nesslerization	4500–NH ₃ D–1997 or E–1997.	D1426–08 (A).	973.49 ³ , I–3520–85. ²
	Titration	4500–NH ₃ F–1997.	D1426–08 (B).	
	Electrode		See footnote. ⁶⁰
5. Antimony—Total, ⁴ mg/L.	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.	350.1 ³⁰ , Rev. 2.0 (1993).	4500–NH ₃ G–1997 4500–NH ₃ H–1997.	I–4523–85. ²
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.		
	Automated electrode	Ion Chromatography.	D6919–09 ..	See footnote. ⁷
	Digestion, ⁴ followed by any of the following:		
	AA direct aspiration ³⁶	3111 B–1999.		
6. Arsenic—Total, ⁴ mg/L.	AA furnace	3113 B–2004.		
	STGFAA	200.9, Rev. 2.2 (1994).	3120 B–1999.	D1976–07 ..	I–4471–97. ⁵⁰
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3125 B–2009.	D5673–05 ..	993.14, ³ I–4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3114 B–2009 or. 3114 C–2009.	D2972–08 (B).	I–3062–85. ²
	Digestion, ⁴ followed by any of the following:	206.5 (Issued 1978) ¹ .	3113 B–2004.	D2972–08 (C).	I–4063–98. ⁴⁹
	AA gaseous hydride		
	AA furnace		
	STGFAA	200.9, Rev. 2.2 (1994).	3120 B–1999.	D1976–07.	
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3125 B–2009.	D5673–05 ..	993.14, ³ I–4020–05. ⁷⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3500–As B–1997.	D2972–08 (A).	I–3060–85. ²
7. Barium—Total, ⁴ mg/L.	Digestion ⁴ , followed by any of the following:		

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/Other
8. Beryllium—Total, ⁴ mg/L.	AA direct aspiration ³⁶	3111 D—1999.	I-3084-85. ²
	AA furnace	3113 B—2004.	D4382—02(07).	
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B—1999.	I-4471-97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B—2009.	D5673-05 ..	993.14, ³ I-4471-97. ⁵⁰
	DCP ³⁶	See footnote. ³⁴
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration	3111 D—1999 or. 3111 E—1999.	D3645-08 (A).	I-3095-85. ²
	AA furnace	3113 B—2004.	D3645-08 (B).	
	STGFAA	200.9, Rev. 2.2 (1994).		
	ICP/AES	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B—1999.	D1976-07 ..	I-4471-97. ⁵⁰
9. Biochemical oxygen demand (BOD ₅), mg/L.	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B—2009.	D5673-05 ..	993.14, ³ I-4471-97. ⁵⁰
	DCP	D4190-08 ..	See footnote. ³⁴
10. Boron—Total, ³⁷ mg/L.	Colorimetric (aluminon)	See foot-note ⁶¹ .		
	Dissolved Oxygen Depletion	5210 B—2001.	973.44 ³ , p. 17. ⁹ , I-1578-78. ⁸
	Colorimetric (curcumin)		See foot-note. ^{10,63}
	ICP/AES	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	4500-B B—2000.	I-3112-85. ²
11. Bromide, mg/L.	ICP/MS	200.8, Rev. 5.4 (1994).	3120 B—1999.	D1976-07 ..	I-4471-97. ⁵⁰
	DCP	D4190-08 ..	See footnote. ³⁴
	Electrode	D1246-05 ..	I-1125-85. ²
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997).	4110 B—2000, C—2000, D—2000.	D4327-03 ..	993.30. ³
12. Cadmium—Total, ⁴ mg/L.	CIE/UV	4140 B—1997.	D6508—00(05).	D6508, Rev. 2. ⁵⁴
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶	3111 B—1999. or 3111 C—1999.	D3557—02(07) (A or B).	974.27, ³ p. 37. ⁹ , I-3135-85. ² or I-3136-85. ²
	AA furnace	3113 B—2004.	D3557—02(07) (D).	I-4138-89. ⁵¹
	STGFAA	200.9, Rev. 2.2 (1994).		
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B—1999.	D1976-07 ..	I-1472-85. ² or I-4471-97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B—2009.	D5673-05 ..	993.14, ³ I-4471-97. ⁵⁰
	DCP ³⁶	D4190-08 ..	See footnote. ³⁴
	Voltammetry ¹¹	D3557—02(07) (C).	

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/Other
13. Calcium—Total, ⁴ mg/L.	Colorimetric (Dithizone) Digestion, ⁴ followed by any of the following: AA direct aspiration ICP/AES ICP/MS DCP Titrimetric (EDTA) 200.5, Rev 4.2 (2003) ⁶⁸ , 200.7, Rev. 4.4 (1994). 200.8, Rev. 5.4 (1994). 3500—Ca B— 1997.	3500—Cd-D— 1990. 3111 B— 1999. 3120 B— 1999. 3125 B— 2009. 5210 B— 2001.	D511—08(B) D5673—05 ..	I—3152—85. ² I—4471—97. ⁵⁰ 993.14. ³ See footnote. ³⁴ See footnote. ^{35,63}
14. Carbo-naceous bio-chemical oxygen demand (CBOD ₅), mg/L ¹² .	Ion Chromatography Dissolved Oxygen Depletion with nitrification inhibitor.	5210 B— 2001.	D6919—09.	
15. Chemical oxygen demand (COD), mg/L.	Titrimetric Spectrophotometric, manual or automatic.	410.3 (Rev. 1978) ¹ . 410.4, Rev. 2.0 (1993).	5220 B— 1997. or C—1997 .. 5220 D— 1997.	D1252—06 (A). D1252—06 (B).	973.46, ³ p. 17, ⁹ I—3560—85. ² See footnotes. ^{13,14} I— 3561—85. ²
16. Chloride, mg/L.	Titrimetric: (silver nitrate) (Mercuric nitrate) Colorimetric: manual Automated (Ferricyanide) Potentiometric Titration Ion Selective Electrode Ion Chromatography CIE/UV 300.0, Rev 2.1 (1993) and 300.1—1, Rev 1.0 (1997).	4500—Cl ⁻ B—1997. 4500—Cl ⁻ C—1997. 4500—Cl ⁻ E—1997. 4500—Cl ⁻ D—1997. 4110 B— 2000 or. 4110 C— 2000. 4140 B— 1997.	D512—04 (B) D512—04 (A) D512—04 (C). D4327—03 ..	973.51, ³ I—1184— 85. ² I—1187—85. ² I—2187—85. ² 993.30 ³ , I— 2057—90. ⁵¹
17. Chlorine—Total residual, mg/L.	Amperometric direct Amperometric direct (low level) Iodometric direct Back titration ether end-point ¹⁵	4500—Cl D— 2000. 4500—Cl E— 2000. 4500—Cl B— 2000. 4500—Cl C— 2000.	D6508— 00(05). D1253—08.	D6508, Rev. 2. ⁵⁴
17A. Chlorine—Free Available, mg/L.	DPD—FAS Spectrophotometric, DPD Electrode Amperometric direct 4500—Cl D— 2000.	4500—Cl F— 2000. 4500—Cl G— 2000. 4500—Cl D— 2000. D1253—08.	See footnote. ¹⁶
18. Chromium VI dissolved, mg/L.	0.45-micron Filtration followed by any of the following:	4500—Cl E— 2000. 4500—Cl F— 2000. 4500—Cl G— 2000.		

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/Other
19. Chromium—Total, ⁴ mg/L.	AA chelation-extraction	3111 C—1999.	I-1232-85. ²
	Ion Chromatography	218.6, Rev. 3.3 (1994).	3500-Cr C—2009.	D5257-03 ..	993.23.
	Colorimetric (Diphenyl-carbazide)	3500-Cr B—2009.	D1687—02(07) (A).	I-1230-85. ²
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶	3111 B—1999.	D1687—02(07) (B).	974.27, ³ I-3236-85. ²
	AA chelation-extraction	3111 C—1999.		
	AA furnace	3113 B—2004.	D1687—02(07) (C).	I-3233-93. ⁴⁶
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B—1999.	D1976-07 ..	I-4471-97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B—2009.	D5673-05 ..	993.14, ³ I-4020-05. ⁷⁰
20. Cobalt—Total, ⁴ mg/L.	DCP ³⁶	3500-Cr B—2009.	D4190-08 ..	See footnote, ³⁴
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration	3111 B—1999 or 3111 C—1999.	D3558-08 (A or B).	p. 37, ⁹ I-3239-85. ²
	AA furnace	3113 B—2004.	D3558-08 (C).	I-4243-89. ⁵¹
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B—1999.	D1976-07 ..	I-4471-97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B—2009.	D5673-05 ..	993.14, ³ I-4020-05. ⁷⁰
	DCP	D4190-08 ..	See footnote, ³⁴
	Colorimetric (ADMI)	See footnote, ¹⁸
	(Platinum cobalt)	2120 B—2001.	I-1250-85. ²
21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity.	Spectrophotometric.				
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶	3111 B—1999 or. 3111 C—1999.	D1688-07 (A or B).	974.27, ³ p. 37, ⁹ I-3270-85 ² or I-3271-85 ²
	AA furnace	3113 B—2004.	D1688-07 (C).	I-4274-89. ⁵¹
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B—1999.	D1976-07 ..	I-4471-97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B—2009.	D5673-05 ..	993.14, ³ I-4020-05. ⁷⁰
	DCP ³⁶	3500-Cu B—1999.	D4190-08 ..	See footnote, ³⁴
	Colorimetric (Neocuproine)	

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/Other
23. Cyanide—Total, mg/L.	(Bathocuproine)	3500—Cu C—1999.	See footnote. ¹⁹
	Automated UV digestion/distillation and Colorimetry.	D7511—09.	Kelada—01. ⁵⁵
	Segmented Flow Injection, In-Line Ultraviolet Digestion, followed by gas diffusion amperometry.	
	Manual distillation with MgCl ₂ , followed by any of the following:	335.4, Rev. 1.0 (1993) ⁵⁷ .	4500—CN [—] B—1999 or C—1999.	D2036—09(A), D7284—08.	10—204—00—1—X. ⁵⁶
	Flow Injection, gas diffusion amperometry.	D2036—09(A) D7284—08.	p. 22. ⁹
	Titrimetric	4500—CN [—] D—1999.	D2036—09(A).	I—3300—85. ²
	Spectrophotometric, manual	4500—CN [—] E—1999.	D2036—09(A).	
	Semi-Automated ²⁰	335.4, Rev. 1.0 (1993) ⁵⁷	10—204—00—1—X, ⁵⁶ I—4302—85. ²
	Ion Chromatography	D2036—09(A).	
	Ion Selective Electrode	4500—CN [—] F—1999.	D2036—09(A).	
24. Cyanide—Available, mg/L.	Cyanide Amenable to Chlorination (CATC); Manual distillation with MgCl ₂ , followed by Titrimetric or Spectrophotometric.	4500—CN [—] G—1999.	D2036—09(B).	
	Flow injection and ligand exchange, followed by gas diffusion amperometry ⁵⁹	D6888—09 ..	OIA—1677—09. ⁴⁴
	Automated Distillation and Colorimetry (no UV digestion).	Kelada—01. ⁵⁵
24.A Cyanide-Free, mg/L.	Flow Injection, followed by gas diffusion amperometry.	D7237—10 ..	OIA—1677—09. ⁴⁴
	Manual micro-diffusion and colorimetry.	D4282—02.	
25. Fluoride—Total, mg/L.	Manual distillation, ⁶ followed by any of the following:	4500—F [—] B—1997.	
	Electrode, manual	4500—F [—] C—1997.	D1179—04 (B).	
	Electrode, automated	I—4327—85. ²
	Colorimetric, (SPADNS)	4500—F [—] D—1997.	D1179—04 (A).	
	Automated complexone	4500—F [—] E—1997.	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1—1, Rev 1.0 (1997).	4110 B—2000 or C—2000.	D4327—03 ..	993.30. ³
	CIE/UV	4140 B—1997.	D6508—00(05).	D6508, Rev. 2. ⁵⁴
26. Gold—Total, ⁴ mg/L.	Digestion, ⁴ followed by any of the following:	
	AA direct aspiration	3111 B—1999.	
	AA furnace	231.2 (Issued 1978) ¹ .	3113 B—2004.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B—2009.	D5673—05 ..	993.14. ³
	DCP	See footnote. ³⁴
27. Hardness—Total, as CaCO ₃ , mg/L.	Automated colorimetric	130.1 (Issued 1971) ¹	
	Titrimetric (EDTA)	2340 C—1997.	D1126—02(07).	973.52B, ³ I—1338—85. ²
	Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33).	2340 B—1997.	

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/ Other
28. Hydrogen ion (pH), pH units.	Electrometric measurement	4500-H+ B-2000.	D1293-99 (A or B).	973.41, ³ I-1586-85, ²
	Automated electrode	150.2 (Dec. 1982) ¹	See footnote, ²¹ I-2587-85. ²
29. Iridium—Total, ⁴ mg/L.	Digestion, ⁴ followed by any of the following: AA direct aspiration	3111 B-1999.		
	AA furnace	235.2 (Issued 1978) ¹		
	ICP/MS	3125 B-2009.		
30. Iron—Total, ⁴ mg/L.	Digestion, ⁴ followed by any of the following: AA direct aspiration ³⁶	3111 B-1999 or. 3111 C-1999.	D1068-05 (A or B).	974.27, ³ I-3381-85, ²
	AA furnace	3113 B-2004.	D1068-05 (C).	
	STGFAA	200.9, Rev. 2.2 (1994).		
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B-1999.	D1976-07 ..	I-4471-97, ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B-2009.	D5673-05 ..	993.14. ³
	DCP ³⁶	D4190-08 ..	See footnote, ³⁴
	Colorimetric (Phenanthroline)	3500-Fe-1997.	D1068-05 (D). D3590-02(06) (A).	See footnote, ²² I-4515-91. ⁴⁵
31. Kjeldahl Nitrogen ⁵ —Total, (as N), mg/L.	Manual digestion ²⁰ and distillation or gas diffusion, followed by any of the following: Titration	4500-N _{org} B-1997 or C-1997 and 4500-NH ₃ B-1997.	
	Nesslerization	4500-NH ₃ C-1997.	973.48. ³
	Electrode	4500-NH ₃ D-1997 or E-1997.	D1426-08 (A). D1426-08 (B).	
	Semi-automated phenate	350.1 Rev 2.0 1993.	4500-NH ₃ G-1997. 4500-NH ₃ H-1997. 4500-NH ₃ F-1997.	
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.	4500-NH ₃ F-1997.	See footnote, ⁶⁰
Automated Methods for TKN that do not require manual distillation					
32. Lead—Total, ⁴ mg/L.	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation). Semi-automated block digester colorimetric (distillation not required). Block digester, followed by Auto distillation and Titration. Block digester, followed by Auto distillation and Nesslerization. Block Digester, followed by Flow injection gas diffusion (distillation not required). Digestion, ⁴ followed by any of the following:	351.1 (Rev. 1978) ¹ . 351.2, Rev. 2.0 (1993). 4500-N _{org} D-1997. D3590-02(06) (B).	I-4551-78, ⁸ I-4515-91. ⁴⁵ See footnote, ³⁹ See footnote, ⁴⁰ See footnote, ⁴¹

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/ Other
33. Magnesium— Total, ⁴ mg/L.	AA direct aspiration ³⁶	3111 B— 1999 or. 3111 C— 1999.	D3559–08 (A or B).	974.27, ³ I–3399– 85, ²
	AA furnace	3113 B— 2004.	D3559–08 (D).	I–4403–89, ⁵¹
	STGFAA	200.9, Rev. 2.2 (1994).
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B— 1999.	D1976–07 ..	I–4471–97, ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B— 2009.	D5673–05 ..	993.14, ³ I–4471– 97, ⁵⁰
	DCP ³⁶	D4190–08 ..	See footnote, ³⁴
	Voltammetry ¹¹	D3559–08 (C).
	Colorimetric (Dithizone)	3500–Pb B— 1997.
	Digestion, ⁴ followed by any of the following:
	AA direct aspiration	3111 B— 1999.	D511–08 (B)	974.27, ³ I–3447– 85, ²
34. Manganese— Total, ⁴ mg/L.	ICP/AES	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B— 1999.	D1976–07 ..	I–4471–97, ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B— 2009.	D5673–05 ..	993.14, ³
	DCP	See footnote, ³⁴
	Gravimetric,
	Ion Chromatography	D6919–09.
	Digestion ⁴ followed by any of the following:
	AA direct aspiration ³⁶	3111 B— 1999.	D858–07 (A or B), D858–07 (C).	974.27, ³ I–3454– 85, ²
	AA furnace	3113 B— 2004.
	STGFAA	200.9, Rev. 2.2 (1994).
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B— 1999.	D1976–07 ..	I–4471–97, ⁵⁰
35. Mercury— Total, ⁴ mg/L.	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B— 2009.	D5673–05 ..	993.14, ³ I–4471– 97, ⁵⁰
	DCP ³⁶	D4190–08 ..	See footnote, ³⁴
	Colorimetric (Persulfate)	3500–Mn B— 1999.	920.203, ³
	(Periodate)	See footnote, ²³
	Cold vapor, Manual	245.1, Rev. 3.0 (1994).	3112 B— 2009.	D3223– 02(07).	977.22, ³ I–3462– 85, ²
36. Molyb- denum—Total, ⁴ mg/L.	Cold vapor, Automated	245.2 (Issued 1974) ¹
	Cold vapor atomic fluorescence spec- trometry (CVAFS).	245.7 Rev. 2.0 (2005) ¹⁷	I–4464–01, ⁷¹
	Purge and Trap CVAFS	1631E ⁴³
	Digestion, ⁴ followed by any of the following:
	AA direct aspiration	3111 D— 1999.	I–3490–85, ²
	AA furnace	3113 B— 2004.	I–3492–96, ⁴⁷
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B— 1999.	D1976–07 ..	I–4471–97, ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B— 2009.	D5673–05 ..	993.14, ³ I–4471– 97, ⁵⁰
	DCP	See footnote, ³⁴

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/ Other
37. Nickel— Total, ⁴ mg/L.	Digestion ⁴ followed by any of the following: AA direct aspiration ³⁶	3111 B– 1999 or. 3111 C– 1999.	D1886–08 (A or B).	I–3499–85. ²
	AA furnace	3113 B– 2004.	D1886–08 (C).	I–4503–89. ⁵¹
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003) ⁶⁸ , 200.7, Rev. 4.4 (1994).	3120 B– 1999.	D1976–07 ..	I–4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B– 2009.	D5673–05 ..	993.14, ³ I–4020– 05. ⁷⁰
	DCP ³⁶			D4190–08 ..	See footnote. ³⁴
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1–1, Rev 1.0 (1997).	4110 B– 2000 or C–2000.	D4327–03 ..	993.30. ³
38. Nitrate (as N), mg/L.	CIE/UV	4140 B– 1997.	D6508– 00(05).	D6508, Rev. 2. ⁵⁴
	Ion Selective Electrode	4500–NO ₃ [–] D–2000.		
	Colorimetric (Brucine sulfate)	352.1 (Issued 1971) ¹		973.50, ⁹ 419D ^{1,7} , p. 28. ⁹
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).				See footnote. ⁶²
	Cadmium reduction, Manual	4500–NO ₃ [–] E–2000.	D3867–04 (B).	
39. Nitrate-nitrite (as N), mg/L.	Cadmium reduction, Automated ..	353.2, Rev. 2.0 (1993).	4500–NO ₃ [–] F–2000.	D3867–04 (A).	I–2545–90. ⁵¹
	Automated hydrazine	4500–NO ₃ [–] H–2000.		
	Reduction/Colorimetric				
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1–1, Rev 1.0 (1997).	4110 B– 2000 or C–2000.	D4327–03 ..	See footnote. ⁶² 993.30. ³
	CIE/UV	4140 B– 1997.	D6508– 00(05).	D6508, Rev. 2. ⁵⁴
40. Nitrite (as N), mg/L.	Spectrophotometric: Manual	4500–NO ₂ [–] B–2000.		See footnote. ²⁵
	Automated (Diazotization)			I–4540–85 ² , See footnote. ⁶²
	Automated (*bypass cadmium re- duction).	353.2, Rev. 2.0 (1993).	4500–NO ₃ [–] F–2000.	D3867–04 (A).	I–4545–85. ²
	Manual (*bypass cadmium reduc- tion).	4500–NO ₃ [–] E–2000.	D3867–04 (B).	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1–1, Rev 1.0 (1997).	4110 B– 2000 or C–2000.	D4327–03 ..	993.30. ³
41. Oil and grease—Total recoverable, mg/L.	CIE/UV	4140 B– 1997.	D6508– 00(05).	D6508, Rev. 2. ⁵⁴
	Hexane extractable material (HEM): n–Hexane extraction and gravim- etry.	1664 Rev. A; 1664 Rev. B ⁴² .	5520 B– 2001 ³⁸ .		
	Silica gel treated HEM (SGT– HEM): Silica gel treatment and gravimetry.	1664 Rev. A; 1664 Rev. B ⁴² .	5520 B– 2001 ³⁸ and 5520 F–2001 ³⁸ .		
42. Organic car- bon—Total (TOC), mg/L.	Combustion	5310 B– 2000.	D7573–09 ..	973.47 ³ , p. 14. ²⁴
	Heated persulfate or UV persulfate oxidation.	5310 C 2000. 5310 D 2000.	D4839–03 ..	973.47 ³ , p. 14. ²⁴

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Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/Other
43. Organic nitrogen (as N), mg/L.	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4).				
44. Ortho-phosphate (as P), mg/L.	Ascorbic acid method: Automated	365.1, Rev. 2.0 (1993).	4500-P F—1999 or G—1999.	973.56 ³ , I-4601-85. ²
	Manual single reagent	4500-P E—1999.	D515-88(A)	973.55. ³
	Manual two reagent	365.3 (Issued 1978) ¹ .			
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997).	4110 B—2000 or C—2000.	D4327-03 ..	993.30. ³
	CIE/UV	4140 B—1997.	D6508—00(05).	D6508, Rev. 2. ⁵⁴
45. Osmium—Total ⁴ , mg/L.	Digestion ⁴ , followed by any of the following: AA direct aspiration,	3111 D—1999.		
	AA furnace	252.2 (Issued 1978) ¹ .			
46. Oxygen, dissolved, mg/L.	Winkler (Azide modification)	4500-O B—2001, C—2001, D—2001, E—2001, F—2001.	D888-09 (A)	973.45B ³ , I-1575-78. ⁸
	Electrode	4500-O G—2001.	D888-09 (B)	I-1576-78. ⁸
	Luminescence Based Sensor	D888-09 (C).	See footnote ⁶³ See footnote ⁶⁴
47. Palladium—Total, ⁴ mg/L.	Digestion ⁴ , followed by any of the following: AA direct aspiration	3111 B—1999.		
	AA furnace	253.2 ¹ (Issued 1978).			
	ICP/MS	3125 B—2009.		
	DCP		See footnote ³⁴
48. Phenols, mg/L.	Manual distillation ²⁶ , followed by any of the following: Colorimetric (4AAP) manual	420.1 ¹ (Rev. 1978).	5530 B—2005.	D1783-01.	
	Automated colorimetric (4AAP) ...	420.1 ¹ (Rev. 1978).	5530 D—2005 ²⁷ .	D1783-01 (A or B).	
49. Phosphorus (elemental), mg/L.	Gas-liquid chromatography	420.4 Rev. 1.0 (1993).	See footnote ²⁸
50. Phosphorus—Total, mg/L.	Digestion ²⁰ , followed by any of the following: Manual	365.3 ¹ (Issued 1978).	4500-P B(5)-1999.	973.55. ³
	Automated ascorbic acid reduction.	365.1 Rev. 2.0 (1993).	4500-P E—1999.	D515-88 (A).	
	ICP/AES ^{4, 36}	200.7, Rev. 4.4 (1994).	4500-P F—1999, G—1999, H—1999.	973.56 ³ , I-4600-85. ²
	Semi-automated block digestor (TKP digestion).	365.4 ¹ (Issued 1974).	3120 B—1999.	I-4471-97. ⁵⁰
51. Platinum—Total, ⁴ mg/L.	Digestion ⁴ followed by any of the following: AA direct aspiration	3111 B—1999.	D515-88 (B)	I-4610-91. ⁴⁸
	AA furnace	255.2 (Issued 1978) ¹ .			

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Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/ Other
52. Potassium—Total, ⁴ mg/L.	ICP/MS DCP Digestion ⁴ , followed by any of the following: AA direct aspiration ICP/AES ICP/MS Flame photometric Electrode Ion Chromatography 200.7, Rev. 4.4 (1994). 200.8, Rev. 5.4 (1994). Gravimetric, 103–105°	3125 B– 2009. 3111 B– 1999. 3120 B– 1999. 3125 B– 2009. 3500-K B– 1997. 3500-K C– 1997. 2540 B– 1997. 2540 C– 1997. 2540 D– 1997. 2540 F– 1997. 2540-E– 1997. 3111 B– 1999. 265.2 (Issued 1978) ¹ . 3125 B– 2009. D5673–05 ..	See footnote, ³⁴ 973.53 ³ , I–3630– 85. ² 993.14. ³
53. Residue—Total, mg/L.	Gravimetric, 180°	2540 B– 1997.	D5907–03 ..	I–3750–85. ²
54. Residue—filterable, mg/L.	Gravimetric, 103–105° post washing of residue.	2540 C– 1997.	D5907–03 ..	I–1750–85. ²
55. Residue—non-filterable (TSS), mg/L.	Volumetric, (Imhoff cone), or gravimetric.	2540 D– 1997.	I–3765–85. ²
56. Residue—settleable, mg/L.	Gravimetric, 550°	160.4 (Issued 1971) ¹ .	2540-E– 1997.	I–3753–85. ²
57. Residue—Volatile, mg/L.	Digestion ⁴ followed by any of the following: AA direct aspiration, or	3111 B– 1999.	
58. Rhodium—Total, ⁴ mg/L.	AA furnace ICP/MS	265.2 (Issued 1978) ¹	265.2 (Issued 1978) ¹ . 3125 B– 2009.	
59. Ruthenium—Total, ⁴ mg/L.	Digestion ⁴ followed by any of the following: AA direct aspiration, or	3111 B– 1999.	
	AA furnace ICP/MS	267.21.	3111 B– 1999. 3125 B– 2009.	
60. Selenium—Total, ⁴ mg/L.	Digestion ⁴ , followed by any of the following: AA furnace	3113 B– 2004.	D3859–08 (B).	I–4668–98. ⁴⁹
	STGFAA	200.9, Rev. 2.2 (1994).	3120 B– 1999.	D1976–07.	
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B– 1999.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B– 2009.	D5673–05 ..	993.14 ³ , I–4020– 05. ⁷⁰
	AA gaseous hydride	3114 B– 2009, or 3111 C– 2009.	3114 B– 2009, or 3111 C– 2009.	D3859–08 (A).	I–3667–85. ²
61. Silica—Dissolved, ³⁷ mg/L.	0.45-micron filtration followed by any of the following: Colorimetric, Manual	4500-SiO ₂ C–1997.	D859–05	I–1700–85. ²
	Automated (Molybdate)	4500-SiO ₂ E–1997 or F–1997.	I–2700–85. ²
	ICP/AES	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B– 1999.	I–4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B– 2009.	D5673–05 ..	993.14. ³

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Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/ Other
62. Silver— Total, ^{4, 31} mg/L.	Digestion ^{4, 29} , followed by any of the following: AA direct aspiration	3111 B– 1999 or 3111 C– 1999.	974.27 ³ , p. 37 ⁹ , I–3720–85. ²
	AA furnace	3113 B– 2004.	I–4724–89. ⁵¹
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.5, Rev. 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B– 1999.	D1976–07 ..	I–4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B– 2009.	D5673–05 ..	993.14 ³ , I–4471– 97. ⁵⁰ See footnote. ³⁴
	DCP	
63. Sodium— Total, ⁴ mg/L.	Digestion ⁴ , followed by any of the following: AA direct aspiration	3111 B– 1999.	973.54 ³ , I–3735– 85. ²
	ICP/AES	200.5, Rev. 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B– 1999.	I–4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B– 2009.	D5673–05 ..	993.14. ³
	DCP	3500–Na B– 1997.	See footnote. ³⁴
	Flame photometric	
64. Specific conductance, micromhos/cm at 25 °C.	Ion Chromatography	D6919–09.	
	Wheatstone bridge	120.1'(Rev. 1982).	2510 B– 1997.	D1125– 95(99) (A).	973.40 ³ , I–2781– 85. ²
65. Sulfate (as SO ₄), mg/L.	Automated colorimetric	375.2, Rev. 2.0 (1993).	4500– SO ₄ ^{2–} F–1997 or G–1997.		
	Gravimetric	4500– SO ₄ ^{2–} C–1997 or D–1997.	925.54. ³
	Turbidimetric	4500– SO ₄ ^{2–} E–1997.	D516–07.	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1–1, Rev. 1.0 (1997).	4110 B– 2000 or C–2000.	D4327–03 ..	993.30 ³ , I–4020– 05. ⁷⁰
	CIE/UV	4140 B– 1997.	D6508– 00(05).	D6508, Rev. 2. ⁵⁴
66. Sulfide (as S), mg/L.	Sample Pretreatment	4500–S ^{2–} B, C– 2000.		
	Titrimetric (iodine)	4500– S ^{2–} F– 2000.	I–3840–85. ²
	Colorimetric (methylene blue)	4500– S ^{2–} D– 2000.		
	Ion Selective Electrode	4500– S ^{2–} G– 2000.	D4658–08.	
67. Sulfite (as SO ₃), mg/L.	Titrimetric (iodine-iodate)	4500– SO ₃ ^{2–} B– 2000.		
68. Surfactants, mg/L.	Colorimetric (methylene blue)	5540 C– 2000.	D2330–02.	
69. Temperature, °C.	Thermometric	2550 B– 2000.	See footnote. ³²

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/ Other
70. Thallium—Total, ⁴ mg/L.	Digestion ⁴ , followed by any of the following: AA direct aspiration	3111 B–1999.		
	AA furnace	279.21(Issued 1978).	3113 B–2004.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.7, Rev. 4.4 (1994); 200.5 Rev. 4.2 (2003) ⁶⁸ .	3120 B–1999.	D1976–07.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2009.	D5673–05 ..	993.14 ³ , I–4471–97. ⁵⁰
71. Tin—Total, ⁴ mg/L.	Digestion ⁴ , followed by any of the following: AA direct aspiration	3111 B–1999.	I–3850–78. ⁸
	AA furnace	3113 B–2004.		
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES	200.5, Rev. 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).			
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2009.	D5673–05 ..	993.14. ³
72. Titanium—Total, ⁴ mg/L.	Digestion ⁴ followed by any of the following: AA direct aspiration	3111 D–1999.		
	AA furnace	283.21(Issued 1978).			
	ICP/AES	200.7, Rev. 4.4 (1994).			
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2009.	D5673–05 ..	993.14. ³
73. Turbidity, NTU ⁵³ .	DCP Nephelometric	180.1, Rev. 2.0 (1993).	2130 B–2001.	D1889–00 ..	See footnote. ³⁴ I–3860–85. ² See footnote. ⁶⁵ See footnote. ⁶⁶ See footnote. ⁶⁷
74. Vanadium—Total, ⁴ mg/L.	Digestion ⁴ , followed by any of the following: AA direct aspiration	3111 D–1999.		
	AA furnace	3113 B–2004.	D3373–03(07).	
	ICP/AES	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B–1999.	D1976–07 ..	I–4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2009.	D5673–05 ..	993.14 ³ , I–4020–05. ⁷⁰
	DCP	3500–V B–1997.	D4190–08 ..	See footnote. ³⁴
	Colorimetric (Gallic Acid)			
75. Zinc—Total ⁴ , mg/L.	Digestion ⁴ , followed by any of the following: AA direct aspiration ³⁶	3111 B–1999 or 3111 C–1999.	D1691–02(07) (A or B).	974.27 ³ , p. 37 ⁹ , I–3900–85. ²
	AA furnace	289.21(Issued 1978).			
	ICP/AES ³⁶	200.5, Rev 4.2 (2003) ⁶⁸ ; 200.7, Rev. 4.4 (1994).	3120 B–1999.	D1976–07 ..	I–4471–97. ⁵⁰

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods	ASTM	USGS/AOAC/Other
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2009.	D5673–05 ..	993.14 ³ , I–4020–05. ⁷⁰
	DCP ³⁶	D4190–08 ..	See footnote ³⁴
	Colorimetric (Zincon)	3500 Zn B–1997.	See footnote ³³
76. Acid Mine Drainage.	1627 ⁶⁹ .			

Table IB Notes:

¹ Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020. Revised March 1983 and 1979, where applicable. U.S. EPA.

² Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Book 5, Chapter A1., unless otherwise stated. 1989. USGS.

³ Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision, 1998. AOAC International.

⁴ For the determination of total metals (which are equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic-metal complexes (to convert the analyte to a detectable form for colorimetric analysis). For non-platform graphite furnace atomic absorption determinations a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness. For direct aspiration flame atomic absorption determinations (FLAA) a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. The approved total recoverable digestion is described as Method 200.2 in Supplement I of "Methods for the Determination of Metals in Environmental Samples" EPA/600R-94/11, May, 1994, and is reproduced in EPA Methods 200.7, 200.8, and 200.9 from the same Supplement. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non-EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instruction and/or cautions. For analyses using inductively coupled plasma-atomic emission spectrometry (ICP-AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (platform furnace AA, ICP-AES, and ICP-MS) use EPA Method 200.2 or an approved alternate procedure (e.g., CEM microwave digestion, which may be used with certain analytes as indicated in Table IB); the total recoverable digestion procedures in EPA Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as "total" metals.

⁵ Copper sulfate or other catalysts that have been found suitable may be used in place of mercuric sulfate.

⁶ Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies. In general, the analytical method should be consulted regarding the need for distillation. If the method is not clear, the laboratory may compare a minimum of 9 different sample matrices to evaluate the need for distillation. For each matrix, a matrix spike and matrix spike duplicate are analyzed both with and without the distillation step. (A total of 36 samples, assuming 9 matrices). If results are comparable, the laboratory may dispense with the distillation step for future analysis. Comparable is defined as < 20% RPD for all tested matrices. Alternatively the two populations of spike recovery percentages may be compared using a recognized statistical test.

⁷ Industrial Method Number 379–75 WE Ammonia, Automated Electrode Method, Technicon Auto Analyzer II. February 19, 1976. Bran & Luebbe Analyzing Technologies Inc.

⁸ The approved method is that cited in Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. USGS.

⁹ American National Standard on Photographic Processing Effluents. April 2, 1975. American National Standards Institute.

¹⁰ In-Situ Method 1003–8–2009, Biochemical Oxygen Demand (BOD) Measurement by Optical Probe. 2009. In-Situ Incorporated.

¹¹ The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

¹² Carbonaceous biochemical oxygen demand (CBOD₅) must not be confused with the traditional BOD₅ test method which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD₅ parameter. A discharger whose permit requires reporting the traditional BOD₅ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD₅ is required can the permittee report data using a nitrification inhibitor.

¹³ OIC Chemical Oxygen Demand Method. 1978. Oceanography International Corporation.

¹⁴ Method 8000, Chemical Oxygen Demand, Hach Handbook of Water Analysis, 1979. Hach Company.

¹⁵ The back titration method will be used to resolve controversy.

¹⁶ Orion Research Instruction Manual, Residual Chlorine Electrode Model 97–70. 1977. Orion Research Incorporated. The calibration graph for the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 mL 0.00281 N potassium iodate/100 mL solution, respectively.

¹⁷ Method 245.7, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, EPA–821–R–05–001. Revision 2.0, February 2005. US EPA.

¹⁸ National Council of the Paper Industry for Air and Stream Improvement (NCASI) Technical Bulletin 253, December 1971.

¹⁹ Method 8506, Biocinchoninate Method for Copper, Hach Handbook of Water Analysis. 1979. Hach Company.

²⁰ When using a method with block digestion, this treatment is not required.

²¹ Industrial Method Number 378–75WA, Hydrogen ion (pH) Automated Electrode Method, Bran & Luebbe (Technicon) Autoanalyzer II. October 1976. Bran & Luebbe Analyzing Technologies.

²² Method 8008, 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water. 1980. Hach Company.

²³ Method 8054, Periodate Oxidation Method for Manganese, Hach Handbook of Wastewater Analysis. 1979. Hach Company.

²⁴ Methods for Analysis of Organic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14. 1987. USGS.

²⁵ Method 8507, Nitrogen, Nitrite-Low Range, Diazotization Method for Water and Wastewater. 1979. Hach Company.

²⁶ Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.

²⁷ The colorimetric reaction must be conducted at a pH of 10.0 ± 0.2.

²⁸ Addison, R.F., and R.G. Ackman. 1970. Direct Determination of Elemental Phosphorus by Gas–Liquid Chromatography, *Journal of Chromatography*, 47(3):421–426.

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²⁹ Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M Na₂S₂O₃ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

³⁰ The use of EDTA decreases method sensitivity. Analysts may omit EDTA or replace with another suitable complexing reagent provided that all method specified quality control acceptance criteria are met.

³¹ For samples known or suspected to contain high levels of silver (e.g., in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogen iodide solution by adding 4.0 mL of concentrated NH₄OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I₂ to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH<7 with NH₄OH. Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100-mL volumetric flask and dilute to volume with water.

³² "Water Temperature—Influential Factors, Field Measurement and Data Presentation," Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1. 1975. USGS.

³³ Method 8009, Zinc Method for Zinc, Hach Handbook of Water Analysis, 1979. Hach Company.

³⁴ Method AES0029, Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes. 1986—Revised 1991. Thermo Jarrell Ash Corporation.

³⁵ In-Situ Method 1004-8—2009, Carbonaceous Biochemical Oxygen Demand (CBOD) Measurement by Optical Probe. 2009. In-Situ Incorporated.

³⁶ Microwave-assisted digestion may be employed for this metal, when analyzed by this methodology. Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals. April 16, 1992. CEM Corporation

³⁷ When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.

³⁸ Only use n-hexane (n-Hexane—85% minimum purity, 99.0% min. saturated C6 isomers, residue less than 1 mg/L) extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Methods 1664 Rev. A and 1664 Rev. B). Use of other extraction solvents is prohibited.

³⁹ Method PAI-DK01, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Titrimetric Detection. Revised December 22, 1994. OI Analytical.

⁴⁰ Method PAI-DK02, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Colorimetric Detection. Revised December 22, 1994. OI Analytical.

⁴¹ Method PAI-DK03, Nitrogen, Total Kjeldahl, Block Digestion, Automated FIA Gas Diffusion. Revised December 22, 1994. OI Analytical.

⁴² Method 1664 Rev. B is the revised version of EPA Method 1664 Rev. A. U.S. EPA. February 1999, Revision A. Method 1664, n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. EPA-821-R-98-002, U.S. EPA. February 2010, Revision B. Method 1664, n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. EPA-821-R-10-001.

⁴³ Method 1631, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. EPA-821-R-02-019, Revision E. August 2002, U.S. EPA. The application of clean techniques described in EPA's Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, EPA-821-R-96-011, are recommended to preclude contamination at low-level, trace metal determinations.

⁴⁴ Method OIA-1677-09, Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA). 2010. OI Analytical.

⁴⁵ Open File Report 00-170, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonium Plus Organic Nitrogen by a Kjeldahl Digestion Method and an Automated Photometric Finish that Includes Digest Cleanup by Gas Diffusion. 2000. USGS.

⁴⁶ Open File Report 93-449, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1993. USGS.

⁴⁷ Open File Report 97-198, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry. 1997.. USGS.

⁴⁸ Open File Report 92-146, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis. 1992. USGS.

⁴⁹ Open File Report 98-639, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace-Atomic Absorption Spectrometry. 1999. USGS.

⁵⁰ Open File Report 98-165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry. 1998. USGS.

⁵¹ Open File Report 93-125, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993.. USGS.

⁵² Unless otherwise indicated, all EPA methods, excluding EPA Method 300.1–1, are published in U.S. EPA. May 1994. Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111; or U.S. EPA. August 1993. Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100. EPA Method 300.1 is US EPA. Revision 1.0, 1997, including errata cover sheet April 27, 1999. Determination of Inorganic Ions in Drinking Water by Ion Chromatography.

⁵³ Styrene-divinyl benzene beads (e.g., AMCO-AEPA-1 or equivalent) and stabilized formazin (e.g., Hach StabCal™ or equivalent) are acceptable substitutes for formazin.

⁵⁴ Method D6508, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte. December 2000. Waters Corp.

⁵⁵ Kelada-01, Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate, EPA 821-B-01-009, Revision 1.2, August 2001. US EPA. Note: A 450-W UV lamp may be used in this method instead of the 550-W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.

⁵⁶ QuikChem Method 10-204-00-1-X, Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis. Revision 2.2, March 2005. Lachat Instruments.

⁵⁷ When using sulfide removal test procedures described in EPA Method 335.4-1, reconstitute particulate that is filtered with the sample prior to distillation.

⁵⁸ Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation “followed by” analysis with a method, approved digestion and/or distillation are required prior to analysis.

⁵⁹ Samples analyzed for available cyanide using OI Analytical method OIA-1677-09 or ASTM method D6888-09 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample filtration to no more than 30 minutes to preclude settling of materials in samples.

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⁶⁰ Analysts should be aware that pH optima and chromophore absorption maxima might differ when phenol is replaced by a substituted phenol as the color reagent in Berthelot Reaction ("phenol-hypochlorite reaction") colorimetric ammonium determination methods. For example when phenol is used as the color reagent, pH optimum and wavelength of maximum absorbance are about 11.5 and 635 nm, respectively—see, Patton, C.J. and S.R. Crouch. March 1977. *Anal. Chem.* 49:464–469. These reaction parameters increase to pH > 12.6 and 665 nm when salicylate is used as the color reagent—see, Krom, M.D. April 1980. *The Analyst* 105:305–316.

⁶¹ If atomic absorption or ICP instrumentation is not available, the alumino colorimetric method detailed in the 19th Edition of *Standard Methods* may be used. This method has poorer precision and bias than the methods of choice.

⁶² Easy (1-Reagent) Nitrate Method, Revision November 12, 2011. Craig Chinchilla.

⁶³ Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD₅ and cBOD₅. Revision 1.2, October 2011. Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table 1B for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).

⁶⁴ In-Situ Method 1002-8-2009, Dissolved Oxygen (DO) Measurement by Optical Probe. 2009. In-Situ Incorporated.

⁶⁵ Mitchell Method M5331, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Leck Mitchell.

⁶⁶ Mitchell Method M5271, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Leck Mitchell.

⁶⁷ Orion Method AQ4500, Determination of Turbidity by Nephelometry. Revision 5, March 12, 2009. Thermo Scientific.

⁶⁸ EPA Method 200.5, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry, EPA/600/R-06/115. Revision 4.2, October 2003. US EPA.

⁶⁹ Method 1627, Kinetic Test Method for the Prediction of Mine Drainage Quality, EPA-821-R-09-002. December 2011. US EPA.

⁷⁰ Techniques and Methods Book 5-B1, Determination of Elements in Natural-Water, Biota, Sediment and Soil Samples Using Collision/Reaction Cell Inductively Coupled Plasma-Mass Spectrometry, Chapter 1, Section B, Methods of the National Water Quality Laboratory, Book 5, Laboratory Analysis, 2006. USGS.

⁷¹ Water-Resources Investigations Report 01-4132, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Organic Plus Inorganic Mercury in Filtered and Unfiltered Natural Water With Cold Vapor-Atomic Fluorescence Spectrometry, 2001. USGS.

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
1. Acenaphthene	GC	610.	6410 B-2000	D4657-92 (98).	See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...			
2. Acenaphthylene	HPLC	610	6440 B-2000	D4657-92 (98)..	See foot-note ⁹ , p. 27.
	GC	610.			
3. Acrolein	GC	603.	6410 B-2000	D4657-92 (98)..	See foot-note ⁹ , p. 27.
	GC/MS	624 ⁴ , 1624B.			
4. Acrylonitrile	GC	603.	6440B-2000 ..	D4657-92 (98)..	See foot-note ⁹ , p. 27.
	GC/MS	624 ⁴ , 1624B.			
5. Anthracene	GC	610.	6410 B-2000	See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...			
6. Benzene	HPLC	610	6440 B-2000	D4657-92 (98)..	See foot-note ⁹ , p. 27.
	GC	602			
7. Benzidine	GC/MS	624, 1624B ...	6410 B-2000.	See foot-note ³ , p.1.
	Spectro-photometric.			
8. Benzo(a)anthracene	GC/MS	625 ⁵ , 1625B	6410 B-2000	See foot-note ⁹ , p. 27.
	HPLC	605.			
9. Benzo(a)pyrene	GC	610.	6410 B-2000	D4657-92 (98)..	See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...			
10. Benzo(b)fluoranthene	HPLC	610	6440 B-2000	D4657-92 (98)..	See foot-note ⁹ , p. 27.
	GC	610.			
11. Benzo(g,h,i)perylene	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
	HPLC	610			
11. Benzo(g,h,i)perylene	GC	610.			

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TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—
Continued

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
12. Benzo(k)fluoranthene	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
	HPLC	610	6440 B-2000	D4657-92 (98)..	
13. Benzyl chloride	GC	610.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	
14. Butyl benzyl phthalate	HPLC	610	6440 B-2000	D4657-92 (98)..	
	GC	See foot-note ³ , p. 130.
15. bis(2-Chloroethoxy) methane	GC/MS	See foot-note ⁶ , p. S102.
	GC	611.			See foot-note ⁹ , p. 27.
16. bis(2-Chloroethyl) ether	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
	GC	611.			See foot-note ⁹ , p. 27.
17. bis(2-Ethylhexyl) phthalate	GC/MS	606.			See foot-note ⁹ , p. 27.
	GC	625, 1625B ...	6410 B-2000	
18. Bromodichloromethane	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
19. Bromoform	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
20. Bromomethane	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
21. 4-Bromophenyl phenyl ether	GC	611.			
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
22. Carbon tetrachloride	GC	601	6200 C-1997	See foot-note ³ , p. 130.
	GC/MS	624, 1624B ...	6200 B-1997.		
23. 4-Chloro-3-methyl phenol	GC	604	6420 B-2000.		See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000.		
24. Chlorobenzene	GC	601, 602	6200 C-1997	See foot-note ³ , p. 130.
	GC/MS	624, 1624B ...	6200 B-1997.		
25. Chloroethane	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
26. 2-Chloroethylvinyl ether	GC	601.			
	GC/MS	624, 1624B.			
27. Chloroform	GC	601	6200 C-1997	See foot-note ³ , p. 130.
	GC/MS	624, 1624B ...	6200 B-1997.		
28. Chloromethane	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
29. 2-Chloronaphthalene	GC	612.			
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
30. 2-Chlorophenol	GC	604	6420 B-2000.		

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 TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—
 Continued

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
31. 4-Chlorophenyl phenyl ether	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
32. Chrysene	GC	611.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
33. Dibenzo(a,h)anthracene	GC	610.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
	HPLC	610	6440 B-2000	D4657-92 (98)..	
34. Dibromochloromethane	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
35. 1,2-Dichlorobenzene	GC	601, 602	6200 C-1997.		See foot-note ⁹ , p. 27.
	GC/MS	624, 1625B ...	6200 B-1997	
36. 1,3-Dichlorobenzene	GC	601, 602	6200 C-1997.		See foot-note ⁹ , p. 27.
	GC/MS	624, 1625B ...	6200 B-1997	
37. 1,4-Dichlorobenzene	GC	601, 602	6200 C-1997.		See foot-note ⁹ , p. 27.
	GC/MS	624, 1625B ...	6200 B-1997	
38. 3,3'-Dichlorobenzidine	GC/MS	625, 1625B ...	6410 B-2000.		
39. Dichlorodifluoromethane	HPLC	605.			
	GC	601.			
40. 1,1-Dichloroethane	GC	6200 C-1997.		
	GC/MS	601	6200 C-1997.		
41. 1,2-Dichloroethane	GC	624, 1624B ...	6200 B-1997.		
	GC/MS	601	6200 C-1997.		
42. 1,1-Dichloroethene	GC	624, 1624B ...	6200 B-1997.		
	GC/MS	601	6200 C-1997.		
43. trans-1,2-Dichloroethene	GC	624, 1624B ...	6200 C-1997.		
	GC/MS	601	6200 B-1997.		
44. 2,4-Dichlorophenol	GC	604	6420 B-2000.		
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
45. 1,2-Dichloropropane	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
46. cis-1,3-Dichloropropene	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
47. trans-1,3-Dichloropropene	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
48. Diethyl phthalate	GC	606.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	
49. 2,4-Dimethylphenol	GC	604	6420 B-2000.		
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
50. Dimethyl phthalate	GC	606.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	
51. Di-n-butyl phthalate	GC	606.			

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TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—
Continued

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
52. Di-n-octyl phthalate	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
53. 2, 4-Dinitrophenol	GC	606.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
54. 2,4-Dinitrotoluene	GC	604	6420 B-2000	See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
55. 2,6-Dinitrotoluene	GC	609.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
56. Epichlorohydrin	GC	See foot-note ³ , p. 130.
	GC/MS	See foot-note ⁶ , p. S102.
57. Ethylbenzene	GC	602	6200 C-1997.		
	GC/MS	624, 1624B ...	6200 B-1997.		
58. Fluoranthene	GC	610.		.	See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	
59. Fluorene	HPLC	610	6440 B-2000	D4657-92 (98)..	
	GC	610.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	
	HPLC	610	6440 B-2000	D4657-92 (98)..	
60. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran	GC/MS	1613B.			
61. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran	GC/MS	1613B.			
62. 1,2,3,4,6,7,8- Heptachloro-dibenzo-p-dioxin.	GC/MS	1613B.			
63. Hexachlorobenzene	GC	612.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	
64. Hexachlorobutadiene	GC	612.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	
65. Hexachlorocyclopentadiene	GC	612.			See foot-note ⁹ , p. 27.
	GC/MS	625 ⁵ , 1625B	6410 B-2000	
66. 1,2,3,4,7,8-Hexachloro-dibenzofuran	GC/MS	1613B.			
67. 1,2,3,6,7,8-Hexachloro-dibenzofuran	GC/MS	1613B.			
68. 1,2,3,7,8,9-Hexachloro-dibenzofuran	GC/MS	1613B.			
69. 2,3,4,6,7,8-Hexachloro-dibenzofuran	GC/MS	1613B.			
70. 1,2,3,4,7,8-Hexachloro-dibenzo-p-dioxin	GC/MS	1613B.			
71. 1,2,3,6,7,8-Hexachloro-dibenzo-p-dioxin	GC/MS	1613B.			
72. 1,2,3,7,8,9-Hexachloro-dibenzo-p-dioxin	GC/MS	1613B.			
73. Hexachloroethane	GC	612.			
	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
74. Indeno(1,2,3-c,d) pyrene	GC	610.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ...	6410 B-2000	

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 TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—
 Continued

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
75. Isophorone	HPLC	610	6440 B–2000	D4657–92 (98)..	
	GC	609.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ..	6410 B–2000		See foot-note ³ , p. 130.
76. Methylene chloride	GC	601	6200 C–1997.		
77. 2-Methyl-4,6-dinitrophenol	GC/MS	624, 1624B ..	6200 B–1997.		See foot-note ⁹ , p. 27.
	GC	604	6420 B–2000.		
	GC/MS	625, 1625B ..	6410 B–2000.		
78. Naphthalene	GC	610.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ..	6410 B–2000.		
79. Nitrobenzene	HPLC	610	6440 B–2000.		See foot-note ⁹ , p. 27.
	GC	609.			
	GC/MS	625, 1625B ..	6410 B–2000		
80. 2-Nitrophenol	HPLC	D4657–92 (98)..	
	GC	604	6420 B–2000.		See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ..	6410 B–2000		
81. 4-Nitrophenol	GC	604	6420 B–2000.		See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ..	6410 B–2000		
82. N-Nitrosodimethylamine	GC	607.			See foot-note ⁹ , p. 27.
	GC/MS	625 ⁵ , 1625B	6410 B–2000		
83. N-Nitrosodi-n-propylamine	GC	607.			See foot-note ⁹ , p. 27.
	GC/MS	625 ⁵ , 1625B	6410 B–2000		
84. N-Nitrosodiphenylamine	GC	607.			See foot-note ⁹ , p. 27.
	GC/MS	625 ⁵ , 1625B	6410 B–2000		
85. Octachlorodibenzofuran	GC/MS	1613B. ¹⁰			
86. Octachlorodibenzo-p-dioxin	GC/MS	1613B. ¹⁰			
87. 2,2'-Oxybis(2-chloro-propane) [also known as bis(2-Chloroisopropyl) ether].	GC	611.			
88. PCB–1016	GC/MS	625, 1625B ..	6410 B–2000		See foot-note ⁹ , p. 27.
	GC	608	See foot-note ³ , p. 43; See footnote. ⁸
	GC/MS	625	6410 B–2000.		
89. PCB–1221	GC/MS	625	6410 B–2000.		See foot-note ³ , p. 43; See footnote. ⁸
	GC	608	
90. PCB–1232	GC/MS	625	6410 B–2000.		See foot-note ³ , p. 43; See footnote. ⁸
	GC	608	
91. PCB–1242	GC/MS	625	6410 B–2000.		See foot-note ³ , p. 43; See footnote. ⁸
	GC	608	

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TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—
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Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
92. PCB-1248	GC/MS	625	6410 B-2000.		
	GC	608.			
	GC/MS	625	6410 B-2000.		
93. PCB-1254	GC	608			See foot-note ³ , p. 43; See footnote. ⁸
	GC/MS	625	6410 B-2000.		
94. PCB-1260	GC	608			See foot-note ³ , p. 43; See footnote. ⁸
	GC/MS	625	6410 B-2000.		
95. 1,2,3,7,8-Pentachloro-dibenzofuran	GC/MS	625	6410 B-2000.		
96. 2,3,4,7,8-Pentachloro-dibenzofuran	GC/MS	1613B.			
97. 1,2,3,7,8-Pentachloro-dibenzo-p-dioxin	GC/MS	1613B.			
98. Pentachlorophenol	GC	604	6420 B-2000		See foot-note ³ , p. 140.
	GC/MS	625, 1625B ..	6410 B-2000		See foot-note ⁹ , p. 27.
99. Phenanthrene	GC	610.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ..	6410 B-2000		
	HPLC	610	6440 B-2000	D4657-92 (98)..	
100. Phenol	GC	604	6420 B-2000.		See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ..	6410 B-2000		
101. Pyrene	GC	610.			See foot-note ⁹ , p. 27.
	GC/MS	625, 1625B ..	6410 B-2000		
	HPLC	610	6440 B-2000	D4657-92 (98)..	
102. 2,3,7,8-Tetrachloro-dibenzofuran	GC/MS	1613B. ¹⁰			
103. 2,3,7,8-Tetrachloro-dibenzo-p-dioxin	GC/MS	613, 625 ^{5a} , 1613B.			
104. 1,1,2,2-Tetrachloroethane	GC	601	6200 C-1997		See foot-note ³ , p. 130.
	GC/MS	624, 1624B ..	6200 B-1997.		
105. Tetrachloroethene	GC	601	6200 C-1997		See foot-note ³ , p. 130.
	GC/MS	624, 1624B ..	6200 B-1997.		
106. Toluene	GC/MS	624, 1624B ..	6200 B-1997.		
	GC	602	6200 C-1997.		
107. 1,2,4-Trichlorobenzene	GC/MS	624, 1624B ..	6200 B-1997.		See foot-note ³ , p. 130.
	GC	612			
	GC/MS	625, 1625B ..	6410 B-2000		See foot-note ⁹ , p. 27.
108. 1,1,1-Trichloroethane	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ..	6200 B-1997.		
109. 1,1,2-Trichloroethane	GC	601	6200 C-1997.		See foot-note ³ , p. 130.
	GC/MS	624, 1624B ..	6200 B-1997.		
110. Trichloroethene	GC	601	6200 C-1997.		
	GC/MS	624, 1624B ..	6200 B-1997.		
111. Trichlorofluoromethane	GC	601	6200 C-1997.		
	GC/MS	624	6200 B-1997.		
112. 2,4,6-Trichlorophenol	GC	604	6420 B-2000.		

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 TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—
Continued

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
113. Vinyl chloride	GC/MS	625, 1625B ...	6410 B-2000	See foot-note ⁹ , p. 27.
114. Nonylphenol	GC	601	6200 C-1997.		
115. Bisphenol A (BPA)	GC/MS	624, 1624B ...	6200 B-1997.	D7065-06.	
116. p-tert-Octylphenol (OP)	GC/MS	D7065-06.	
117. Nonylphenol Monoethoxylate (NP1EO)	GC/MS	D7065-06.	
118. Nonylphenol Diethoxylate (NP2EO)	GC/MS	D7065-06.	
119. Adsorbable Organic Halides (AOX)	Adsorption and Coulometric Titration.	1650. ¹¹	D7065-06.	
120. Chlorinated Phenolics	In Situ Acetylation and GC/MS.	1653. ¹¹

Table IC notes:

¹ All parameters are expressed in micrograms per liter ($\mu\text{g}/\text{L}$) except for Method 1613B, in which the parameters are expressed in picograms per liter (pg/L).

² The full text of Methods 601–613, 624, 625, 1613B, 1624B, and 1625B are provided at Appendix A, Test Procedures for Analysis of Organic Pollutants, of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, Definition and Procedure for the Determination of the Method Detection Limit, of this Part 136.

³ Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater. September 1978. U.S. EPA.

⁴ Method 624 may be used for quantitative determination of acrolein and acrylonitrile, provided that the laboratory has documentation to substantiate the ability to detect and quantify these analytes at levels necessary to comply with any associated regulations. In addition, the use of sample introduction techniques other than simple purge-and-trap may be required. QC acceptance criteria from Method 603 should be used when analyzing samples for acrolein and acrylonitrile in the absence of such criteria in Method 624.

⁵ Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, N-nitrosodi-n-propylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.

⁶ Method 625, screening only.

⁶ Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the 15th Edition of *Standard Methods for the Examination of Water and Wastewater*. 1981. American Public Health Association (APHA).

⁷ Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624B, and 1625B in accordance with procedures each in Section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 1624B and 1625B) of all samples to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

⁸ Organochlorine Pesticides and PCBs in Wastewater Using EmporeTM Disk. Revised October 28, 1994. 3M Corporation.

⁹ Method O-3116-87 is in Open File Report 93-125, Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. USGS.

¹⁰ Analyses may use Fluid Management Systems, Inc. Power-Prep system in place of manual cleanup provided the analyst meets the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities. Method 1613, Revision B, Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. Revision B, 1994. U.S. EPA. The full text of this method is provided in Appendix A to 40 CFR Part 136 and at <http://water.epa.gov/scitech/methods/cwa/index.cfm>

¹¹ Method 1650, Adsorbable Organic Halides by Adsorption and Coulometric Titration. Revision C, 1997. U.S. EPA. Method 1653, Chlorinated Phenolics in Wastewater by In Situ Acetylation and GCMS. Revision A, 1997. U.S. EPA. The full text for both of these methods is provided at Appendix A in Part 430, The Pulp, Paper, and Paperboard Point Source Category.

 TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
1. Aldrin	GC	608, 617	6630 B–2000 & C–2000.	D3086–90, D5812–96 (02).	See footnote ³ , p. 7; See footnote ⁴ , O-3104–83; See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B–2000.
2. Ametryn	GC	507, 619	See footnote ³ , p. 83; See footnote ⁹ , O-3106–93; See footnote ⁶ , p. S68.
	GC/MS	525.2	See footnote ¹⁴ , O-1121–91.
3. Aminocarb	TLC	See footnote ³ , p. 94; See footnote ⁶ , p. S60.
	HPLC	632.

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TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹—Continued

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
4. Atraton	GC	619	See footnote ³ , p. 83; See footnote ⁶ , p. S68.
5. Atrazine	GC	507, 619	See footnote ³ , p. 83; See footnote ⁶ , p. S68; See footnote ⁹ , O-3106-93.
	HPLC/MS	See footnote ¹² , O-2060-01.
	GC/MS	525.1, 525.2	See footnote ¹¹ , O-1126-95.
6. Azinphos methyl	GC	614, 622, 1657	See footnote ³ , p. 25; See footnote ⁶ , p. S51.
	GC-MS	See footnote ¹¹ , O-1126-95.
7. Barban	TLC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
8. α-BHC	HPLC	632.	See footnote ³ , p. 7; See footnote ⁸ , 3M0222.
	GC	608, 617	6630 B-2000 & C-2000.	D3086-90, D5812-96(02).
	GC/MS	625 ⁵	6410 B-2000.	See footnote ¹¹ , O-1126-95.
9. β-BHC	GC	608, 617	6630 B-2000 & C-2000.	D3086-90, D5812-96(02).	See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B-2000.
10. δ-BHC	GC	608, 617	6630 B-2000 & C-2000.	D3086-90, D5812-96(02).	See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B-2000.
11. γ-BHC (Lindane).	GC	608, 617	6630 B-2000 & C-2000.	D3086-90, D5812-96(02).	See footnote ³ , p. 7; See footnote ⁴ , O-3104-83; See footnote ⁸ , 3M0222.
	GC/MS	625 ⁵	6410 B-2000.	See footnote ¹¹ , O-1126-95.
12. Captan	GC	617	6630 B-2000.	D3086-90, D5812-96(02).	See footnote ³ , p. 7.
13. Carbaryl	TLC	See footnote ³ , p. 94, See footnote ⁶ , p. S60.
	HPLC	531.1, 632.	See footnote ¹² , O-2060-01.
	HPLC/MS	553	See footnote ¹¹ , O-1126-95.
	GC/MS
14. Carbophenothion	GC	617	6630 B-2000.	See footnote ⁴ , page 27; See footnote ⁶ , p. S73.
15. Chlordane	GC	608, 617	6630 B-2000 & C-2000.	D3086-90, D5812-96(02).	See footnote ³ , p. 7; See footnote ⁴ , O-3104-83; See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B-2000.
16. Chlorpropham	TLC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
17. 2,4-D	HPLC	632.	See footnote ³ , p. 115; See footnote ⁴ , O-3105-83.
	GC	615	6640 B-2001.	See footnote ¹² , O-2060-01.
	HPLC/MS
18. 4,4'-DDD	GC	608, 617	6630 B-2000 & C-2000.	D3086-90, D5812-96(02).	See footnote ³ , p. 7; See footnote ⁴ , O-3105-83; See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B-2000.
19. 4,4'-DDE	GC	608, 617	6630 B-2000 & C-2000.	D3086-90, D5812-96(02).	See footnote ³ , p. 7; See footnote ⁴ , O-3104-83; See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B-2000.	See footnote ¹¹ , O-1126-95.

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 TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹—Continued

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
20. 4,4'-DDT	GC	608, 617	6630 B— 2000 & C— 2000.	D3086—90, D5812— 96(02).	See footnote ³ , p. 7; See footnote ⁴ , O—3104—83; See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B— 2000.		
21. Demeton-O	GC	614, 622			See footnote ³ , p. 25; See footnote ⁶ , p. S51.
22. Demeton-S	GC	614, 622			See footnote ³ , p. 25; See footnote ⁶ , p. S51.
23. Diazinon	GC	507, 614, 622, 1657			See footnote ³ , p. 25; See footnote ⁴ , O—3104—83; See footnote ⁶ , p. S51.
	GC/MS	525.2			See footnote ¹¹ , O—1126— 95.
24. Dicamba	GC	615			See footnote ³ , p. 115.
	HPLC/MS				See footnote ¹² , O—2060— 01.
25. Dichlofenthion	GC	622.1			See footnote ⁴ , page 27; See footnote ⁶ , p. S73.
26. Dichloran	GC	608.2, 617	6630 B— 2000.		See footnote ³ , p. 7;
27. Dicofol	GC	617			See footnote ⁴ , O—3104—83.
28. Dieldrin	GC	608, 617	6630 B— 2000 & C— 2000.	D3086—90, D5812— 96(02).	See footnote ³ , p. 7; See footnote ⁴ , O—3104—83; See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B— 2000.		See footnote ¹¹ , O—1126— 95.
29. Dioxathion	GC	614.1, 1657			See footnote ⁴ , page 27; See footnote ⁶ , p. S73.
30. Disulfoton	GC	507, 614, 622, 1657			See footnote ³ , p. 25; See footnote ⁶ , p. S51.
	GC/MS	525.2			See footnote ¹¹ , O—1126— 95.
31. Diuron	TLC				See footnote ³ , p. 104; See footnote ⁶ , p. S64.
	HPLC	632.			See footnote ¹² , O—2060— 01.
32. Endosulfan I	GC	608, 617	6630 B— 2000 & C— 2000.	D3086—90, D5812— 96(02).	See footnote ³ , p. 7; See footnote ⁴ , O—3104—83; See footnote ⁸ , 3M0222.
	GC/MS	625 ⁵	6410 B— 2000.		See footnote ¹³ , O—2002— 01.
33. Endosulfan II	GC	608, 617	6630 B— 2000 & C— 2000.	D3086—90, D5812— 96(02).	See footnote ³ , p. 7; See footnote ⁸ , 3M0222.
	GC/MS	625 ⁵	6410 B— 2000.		See footnote ¹³ , O—2002— 01.
34. Endosulfan Sulfate	GC	608, 617	6630 C— 2000.		See footnote ⁸ , 3M0222.
	GC/MS	625	6410 B— 2000.		
35. Endrin	GC	505, 508, 608, 617, 1656.	6630 B— 2000 & C— 2000.	D3086—90, D5812— 96(02).	See footnote ³ , p. 7; See footnote ⁴ , O—3104—83; See footnote ⁸ , 3M0222.
	GC/MS	525.1, 525.2, 625 ⁵	6410 B— 2000.		
36. Endrin aldehyde	GC	608, 617	6630 C— 2000.		See footnote ⁸ , 3M0222.
37. Ethion	GC/MS	625.			See footnote ⁴ , page 27;
	GC	614, 614.1, 1657			See footnote ⁶ , p. S73.
38. Fenuron	GC/MS				See footnote ¹³ , O—2002— 01.
	TLC				See footnote ³ , p. 104; See footnote ⁶ , p. S64.
39. Fenuron-TCA	HPLC	632.			See footnote ¹² , O—2060— 01.
	HPLC/MS				See footnote ³ , p. 104; See footnote ⁶ , p. S64.

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TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹—Continued

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
40. Heptachlor	HPLC	632.	6630 B— 2000 & C— 2000.	D3086—90, D5812— 96(02).	See footnote ³ , p. 7; See footnote ⁴ , O—3104—83; See footnote ⁸ , 3M0222.
	GC	505, 508, 608, 617, 1656.			
41. Heptachlor epoxide.	GC/MS	525.1, 525.2, 625 ...	6410 B— 2000. 6630 B— 2000 & C— 2000.	D3086—90, D5812— 96(02).	See footnote ³ , p. 7; See footnote ⁴ , O—3104—83; See footnote ⁶ , p. S73; See footnote ⁸ , 3M0222.
	GC	608, 617			
42. Isodrin	GC/MS	625	6410 B— 2000. 6630 B— 2000 & C— 2000.	See footnote ⁴ , O—3104—83; See footnote ⁶ , p. S73.
	GC	617			
43. Linuron	GC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
	HPLC	632.			
44. Malathion	HPLC/MS	553	See footnote ¹² , O—2060— 01. See footnote ¹¹ , O—1126— 95.
	GC/MS			
45. Methiocarb	GC	614, 1657	6630 B— 2000.	See footnote ³ , p. 25; See footnote ⁶ , p. S51. See footnote ¹¹ , O—1126— 95.
	GC/MS			
46. Methoxychlor ...	TLC	See footnote ³ , p. 94; See footnote ⁶ , p. S60.
	HPLC	632.			
47. Mexacarbate ...	HPLC/MS	See footnote ¹² , O—2060— 01. See footnote ³ , p. 7; See footnote ⁴ , O—3104—83; See footnote ⁸ , 3M0222. See footnote ¹¹ , O—1126— 95.
	GC	505, 508, 608.2, 617, 1656.			
48. Mirex	GC/MS	525.1, 525.2	See footnote ³ , p. 94; See footnote ⁶ , p. S60.
	TLC			
49. Monuron	HPLC	632.	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
	GC	617			
50. Monuron-TCA ..	TLC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
	HPLC	632.			
51. Neburon	TLC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
	HPLC	632.			
52. Parathion methyl.	HPLC/MS	See footnote ¹² , O—2060— 01. See footnote ⁴ , page 27; See footnote ³ , p. 25. See footnote ¹¹ , O—1126— 95.
	GC	614, 622, 1657			
53. Parathion ethyl	GC/MS	See footnote ⁴ , page 27; See footnote ³ , p. 25. See footnote ¹¹ , O—1126— 95.
	GC	614			
54. PCNB	GC/MS	See footnote ³ , p. 7.
	GC	608.1, 617			
55. Perthane	GC	617	See footnote ⁴ , O—3104—83.
	GC	507, 619			
56. Prometon	GC	See footnote ³ , p. 83; See footnote ⁶ , p. S68; See footnote ⁹ , O—3106—93.

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 TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹—Continued

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
57. Prometryn	GC/MS	525.2	See footnote ¹¹ , O-1126-95.
	GC	507, 619	See footnote ³ , p. 83; See footnote ⁶ , p. S68; See footnote ⁹ , O-3106-93.
	GC/MS	525.1, 525.2	See footnote ¹³ , O-2002-01.
58. Propazine	GC	507, 619, 1656	See footnote ³ , p. 83; See footnote ⁶ , p. S68; See footnote ⁹ , O-3106-93.
	GC/MS	525.1, 525.2	See footnote ¹³ , O-2002-01.
59. Propham	TLC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
	HPLC	632.	See footnote ¹² , O-2060-01.
60. Propoxur	HPLC/MS	See footnote ³ , p. 94; See footnote ⁶ , p. S60.
	TLC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
61. Secbumeton	HPLC	632.	See footnote ³ , p. 83; See footnote ⁶ , p. S68.
	TLC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
62. Siduron	GC	619.	See footnote ¹² , O-2060-01.
	HPLC	632.	See footnote ³ , p. 83; See footnote ⁶ , p. S68.
63. Simazine	HPLC/MS	See footnote ³ , p. 94; See footnote ⁶ , p. S60.
	GC	505, 507, 619, 1656	See footnote ³ , p. 83; See footnote ⁶ , p. S68; See footnote ⁹ , O-3106-93.
64. Strobane	GC/MS	525.1, 525.2	See footnote ¹¹ , O-1126-95.
	GC	617	6630 B-2000 & C-2000.	See footnote ³ , p. 7.
65. Swep	TLC	See footnote ³ , p. 104; See footnote ⁶ , p. S64.
	HPLC	632.	See footnote ³ , p. 115; See footnote ⁴ , O-3105-83.
66. 2,4,5-T	GC	615	6640 B-2001.	See footnote ³ , p. 115; See footnote ⁴ , O-3105-83.
	GC	615	6640 B-2001.	See footnote ³ , p. 83; See footnote ⁶ , p. S68.
67. 2,4,5-TP (Silvex).	GC	619, 1656	See footnote ¹³ , O-2002-01.
	GC/MS	See footnote ³ , p. 7; See footnote ⁸ ; See footnote ⁴ , O-3105-83.
68. Terbutylazine	GC	505, 508, 608, 617, 1656	6630 B-2000 & C-2000.	D3086-90, D5812-96(02).	See footnote ³ , p. 7; See footnote ⁸ ; See footnote ⁴ , O-3105-83.
	GC/MS	525.1, 525.2, 625 ...	6410 B-2000.	See footnote ³ , p. 7; See footnote ⁹ , O-3106-93.
70. Trifluralin	GC	508, 617, 627, 1656	6630 B-2000.	See footnote ¹¹ , O-1126-95.
	GC/MS	525.2	See footnote ³ , p. 7; See footnote ⁹ , O-3106-93.

Table ID notes:

¹ Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.

² The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, Definition and Procedure for the Determination of the Method Detection Limit, of this Part 136.

³ Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater. September 1978. U.S. EPA. This EPA publication includes thin-layer chromatography (TLC) methods.

⁴ Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3. 1987. USGS.

⁵ The method may be extended to include α -BHC, γ -BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

⁶ Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the 15th Edition of *Standard Methods for the Examination of Water and Wastewater*. 1981. American Public Health Association (APHA).

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⁷ Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

⁸ Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk. Revised October 28, 1994. 3M Corporation.

⁹ Method O-3106-93 is in Open File Report 94-37, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen-Containing Compounds by Gas Chromatography With Nitrogen Phosphorus Detectors. 1994. USGS.

¹⁰ EPA Methods 608.1, 608.2, 614, 614.1, 615, 617, 619, 622, 622.1, 627, and 632 are found in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater. EPA 821-R-92-002, April 1992, U.S. EPA. The full text of Methods 608 and 625 are provided at Appendix A, Test Procedures for Analysis of Organic Pollutants, of this Part 136. EPA Methods 505, 507, 508, 525.1, 531.1 and 553 are in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, 1993, U.S. EPA. EPA Method 525.2 is in Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, Revision 2.0, 1995, U.S. EPA. EPA methods 1656 and 1657 are in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, 1993, U.S. EPA.

¹¹ Method O-1126-95 is in Open-File Report 95-181, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of pesticides in water by C-18 solid-phase extraction and capillary-column gas chromatography/mass spectrometry with selected-ion monitoring. 1995. USGS.

¹² Method O-2060-01 is in Water-Resources Investigations Report 01-4134, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Pesticides in Water by Graphitized Carbon-Based Solid-Phase Extraction and High-Performance Liquid Chromatography/Mass Spectrometry. 2001. USGS.

¹³ Method O-2002-01 is in Water-Resources Investigations Report 01-4098, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of moderate-use pesticides in water by C-18 solid-phase extraction and capillary-column gas chromatography/mass spectrometry. 2001. USGS.

¹⁴ Method O-1121-91 is in Open-File Report 91-519, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of organonitrogen herbicides in water by solid-phase extraction and capillary-column gas chromatography/mass spectrometry with selected-ion monitoring. 1992. USGS.

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TABLE IE—LIST OF APPROVED RADIOLOGIC TEST TEST PROCEDURES

Parameter and units	Method	Reference (method number or page)			
		EPA ¹	Standard Methods 18th, 19th, 20th Ed.	Standard Methods On-line	ASTM
1. Alpha-Total, pCi per liter	Proportional or scintillation counter.	900.0	7110 B	D1943-90, 96	pp. 75 and 78 ³
2. Alpha-Counting error, pCi per liter.	Proportional or scintillation counter.	Appendix B	7110 B	D1943-90, 96	p. 79
3. Beta-Total, pCi per liter	Proportional counter	900.0	7110 B	D1890-90, 96	pp. 75 and 78 ³
4. Beta-Counting error, pCi	Proportional counter	Appendix B	7110 B	D1890-90, 96	p. 79
5. (a) Radium / Total pCi per liter.	Proportional counter	903.0	7500-Ra B	D2460-90, 97	
(b) Ra, pCi per liter	Scintillation counter	903.1	7500-Ra C	D3454-91, 97	p. 81

¹ Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032 (1980), U.S. Environmental Protection Agency, August 1980.

² Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).

³ The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

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TABLE IF—LIST OF APPROVED METHODS FOR PHARMACEUTICAL POLLUTANTS

Pharmaceuticals pollutants	CAS registry No.	Analytical method number
acetonitrile	75-05-8	1666/1671/D3371/D3695.
n-amyl acetate	628-63-7	1666/D3695.
n-amyl alcohol	71-41-0	1666/D3695
benzene	71-43-2	D4763/D3695/502.2/524.2.
n-butyl-acetate	123-86-4	1666/D3695.
tert-butyl alcohol	75-65-0	1666.
chlorobenzene	108-90-7	502.2/524.2.
chloroform	67-66-3	502.2/524.2/551.
o-dichlorobenzene	95-50-1	1625C/502.2/524.2.
1,2-dichloroethane	107-06-2	D3695/502.2/524.2.
diethylamine	109-89-7	1666/1671.
dimethyl sulfoxide	67-68-5	1666/1671.
ethanol	64-17-5	1666/1671/D3695.
ethyl acetate	141-78-6	1666/D3695.
n-heptane	142-82-5	1666/D3695.
n-hexane	110-54-3	1666/D3695.
isobutyraldehyde	78-84-2	1666/1667.
isopropanol	67-63-0	1666/D3695.
isopropyl acetate	108-21-4	1666/D3695.
isopropyl ether	108-20-3	1666/D3695.
methanol	67-56-1	1666/1671/D3695.
Methyl Cellosolve Δ	109-86-4	1666/1671
methylene chloride	75-09-2	502.2/524.2
methyl formate	107-31-3	1666.
4-methyl-2-pentanone (MIBK)	108-10-1	1624C/1666/D3695/D4763/524.2.
phenol	108-95-2	D4763.
n-propanol	71-23-8	1666/1671/D3695.
2-propanone (acetone)	67-64-1	D3695/D4763/524.2.
tetrahydrofuran	109-99-9	1666/524.2.
toluene	108-88-3	D3695/D4763/502.2/524.2.
triethylamine	121-44-8	1666/1671.
xylenes	(Note 1)	1624C/1666.

TABLE 1F NOTE:

1. 1624C: m-xylene 108-38-3, o,p-xylene E-14095 (Not a CAS number; this is the number provided in the Environmental Monitoring Methods Index (EMMI) database.); 1666: m,p-xylene 136777-61-2, o-xylene 95-47-6.

TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS (40 CFR PART 455)

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s) ³
8	Triadimefon	43121-43-3	507/633/525.1/525.2/1656
12	Dichlorvos	62-73-7	1657/507/622/525.1/525.2
16	2,4-D; 2,4-D Salts and Esters [2,4-Dichlorophenoxyacetic acid].	94-75-7	1658/515.1/615/515.2/555
17	2,4-DB; 2,4-DB Salts and Esters [2,4-Dichlorophenoxybutyric acid].	94-82-6	1658/515.1/615/515.2/555
22	Mevinphos	7786-34-7	1657/507/622/525.1/525.2
25	Cyanazine	21725-46-2	629/507
26	Propachlor	1918-16-7	1656/508/608.1/525.1/525.2
27	MCPA; MCPA Salts and Esters [2-Methyl-4-chlorophenoxyacetic acid].	94-74-6	1658/615/555
30	Dichlorprop; Dichlorprop Salts and Esters [2-(2,4-Dichlorophenoxy) propionic acid].	120-36-5	1658/515.1/615/515.2/555
31	MCPP; MCPP Salts and Esters [2-(2-Methyl-4-chlorophenoxy) propionic acid].	93-65-2	1658/615/555
35	TCMTB [2-(Thiocyanomethylthio) benzo-thiazole].	21564-17-0	637
39	Pronamide	23950-58-5	525.1/525.2/507/633.1
41	Propanil	709-98-8	632.1/1656
45	Metribuzin	21087-64-9	507/633/525.1/525.2/1656
52	Acephate	30560-19-1	1656/1657
53	Acifluorfen	50594-66-6	515.1/515.2/555
54	Alachlor	15972-60-8	505/507/645/525.1/525.2/1656
55	Aldicarb	116-06-3	531.1
58	Ametryn	834-12-8	507/619/525.2
60	Atrazine	1912-24-9	505/507/619/525.1/525.2/1656
62	Benomyl	17804-35-2	631
68	Bromacil; Bromacil Salts and Esters	314-40-9	507/633/525.1/525.2/1656
69	Bromoxynil	1689-84-5	1625/1661
69	Bromoxynil octanoate	1689-99-2	1656
70	Butachlor	23184-66-9	507/645/525.1/525.2/1656
73	Captan	2425-06-1	1656

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TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS (40 CFR PART 455)—Continued

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s) ³
75	Carbaryl [Sevin]	63-25-2	531.1/632/553
76	Carbofuran	1563-66-2	531.1/632
80	Chloroneb	2675-77-6	1656/508/608.1/525.1/525.2
82	Chlorothalonil	1897-45-6	508/608.2/525.1/525.2/1656
84	Stirofos	961-11-5	1657/507/622/525.1/525.2
86	Chlorpyrifos	2921-88-2	1657/508/622
90	Fenvalerate	51630-58-1	1660
103	Diazinon	333-41-5	1657/507/614/622/525.2
107	Parathion methyl	298-00-0	1657/614/622
110	DCPA [Dimethyl 2,3,5,6-tetrachloro-terephthalate].	1861-32-1	508/608.2/525.1/525.2/515.1 ² /515.2 ² /1656
112	Dinoseb	88-85-7	1658/515.1/615/515.2/555
113	Dioxathion	78-34-2	1657/614.1
118	Nabonate [Disodium cyanodithio-imidocarbonate].	138-93-2	630.1
119	Diuron	330-54-1	632/553
123	Endothall	145-73-3	548/548.1
124	Endrin	72-20-8	1656/505/508/608/617/525.1/525.2
125	Ethalfuralin	55283-68-6	1656/627 See footnote 1
126	Ethion	563-12-2	1657/614/614.1
127	Ethoprop	13194-48-4	1657/507/622/525.1/525.2
132	Fenarimol	60168-88-9	507/633.1/525.1/525.2/1656
133	Fenthion	55-38-9	1657/622
138	Glyphosate [N-(Phosphonomethyl) glycine]	1071-83-6	547
140	Heptachlor	76-44-8	1656/505/508/608/617/525.1/525.2
144	Isopropalin	33820-53-0	1656/627
148	Linuron	330-55-2	553/632
150	Malathion	121-75-5	1657/614
154	Methamidophos	10265-92-6	1657
156	Methylomyl	16752-77-5	531.1/632
158	Methoxychlor	72-43-5	1656/505/508/608.2/617/525.1/525.2
172	Nabam	142-59-6	630/630.1
173	Naled	300-76-5	1657/622
175	Norflurazon	27314-13-2	507/645/525.1/525.2/1656
178	Benfluralin	1861-40-1	1656/627 See footnote 1
182	Fensulfothion	115-90-2	1657/622
183	Disulfoton	298-04-4	1657/507/614/622/525.2
185	Phosmet	732-11-6	1657/622.1
186	Azinphos Methyl	86-50-0	1657/614/622
192	Organic-tin pesticides	12379-54-3	Ind-01/200.7/200.9
197	Bolstar	35400-43-2	1657/622
203	Parathion	56-38-2	1657/614
204	Pendimethalin	40487-42-1	1656
205	Pentachloronitrobenzene	82-68-8	1656/608.1/617
206	Pentachlorophenol	87-86-5	625/1625/515.2/555/515.1/525.1/525.2
208	Permethrin	52645-53-1	608.2/508/525.1/525.2/1656/1660
212	Phorate	298-02-2	1657/622
218	Busan 85 [Potassium dimethyldithiocarbamate].	128-03-0	630/630.1
219	Busan 40 [Potassium N-hydroxymethyl-N-methyldithiocarbamate].	51026-28-9	630/630.1
220	KN Methyl [Potassium N-methyl-dithiocarbamate].	137-41-7	630/630.1
223	Prometon	1610-18-0	507/619/525.2
224	Prometryn	7287-19-6	507/619/525.1/525.2
226	Propazine	139-40-2	507/619/525.1/525.2/1656
230	Pyrethrin I	121-21-1	1660
232	Pyrethrin II	121-29-9	1660
236	DEF [S,S,S-Tributyl phosphorotri thioate]	78-48-8	1657
239	Simazine	122-34-9	505/507/619/525.1/525.2/1656
241	Carbam-S [Sodium dimethyl dithio-carbamate]	128-04-1	630/630.1
243	Vapam [Sodium methyl dithiocarbamate]	137-42-8	630/630.1
252	Tebuthiuron	34014-18-1	507/525.1/525.2
254	Terbacil	5902-51-2	507/633/525.1/525.2/1656
255	Terbufos	13071-79-9	1657/507/614.1/525.1/525.2
256	Terbutylazine	5915-41-3	619/1656
257	Terbutryn	886-50-0	507/619/525.1/525.2
259	Dazomet	533-74-4	630/630.1/1659
262	Toxaphene	8001-35-2	1656/505/508/608/617/525.1/525.2
263	Merphos [Tributyl phosphorotri thioate]	150-50-5	1657/507/525.1/525.2/622
264	Trifluralin ¹	1582-09-8	1656/508/617/627/525.2

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TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS (40 CFR PART 455)—Continued

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s) ³
268	Ziram [Zinc dimethyldithiocarbamate]	137-30-4	630/630.1

Table 1G notes:

¹ Monitor and report as total Trifluralin.

² Applicable to the analysis of DCPA degradates.

³ EPA Methods 608.1 through 645, 1645 through 1661, and Ind-01 are available in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, Revision I, August 1993, U.S. EPA. EPA Methods 200.9 and 505 through 555 are available in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, August 1993, U.S. EPA. The full text of Methods 608, 625 and 1625 are provided at Appendix A of this Part 136. The full text of Method 200.7 is provided at appendix C of this part 136.

TABLE IH—LIST OF APPROVED MICROBIOLOGICAL METHODS FOR AMBIENT WATER

Parameter and units	Method ¹	EPA	Standard methods	AOAC, ASTM, USGS	Other
Bacteria:					
1. Coliform (fecal), number per 100 mL or number per gram dry weight.	Most Probable Number (MPN), 5 tube, 3 dilution, or. Membrane filter (MF) ² , single step. MPN, 5 tube, 3 dilution, or.	p. 132 ³ p. 124 ³ p. 132 ³	9221 C E-2006. 9222 D-1997 9221 C E-2006.		
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MF ² , single step ⁵	p. 124 ³	9222 D-1997.	B-0050-85 ⁴	
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or. MF ² , single step or two step. MPN, 5 tube, 3 dilution, or.	p. 114 ³ p. 108 ³ p. 114 ³	9221 B-2006. 9222 B-1997. 9221 B-2006.		
4. Coliform (total), in presence of chlorine, number per 100 mL.	MF ² with enrichment	p. 111 ³	9222 (B+B.5c)-1997.	B-0025-85 ⁴	
5. <i>E. coli</i> , number per 100 mL	MPN ^{6,8,14} , multiple tube, or. Multiple tube/multiple well, or. MF ^{2,5,6,7,8} , two step, or 1103.1 ¹⁹	9221 B-1-2006/9221 F-2006 ^{11,13} . 9223 B-2004 ¹² .	991.15 ¹⁰	Colilert ^{®12,16} , Colilert-18 ^{®12,15,16} .
	Single step	1603 ²⁰ , 1604 ²¹	mColiBlue-24 ^{®17} .
6. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution, or. MF ² , or	p. 139 ³ p. 136 ³	9230 B-2007. 9230 C-2007.	B-0055-85 ⁴ .	
7. Enterococci, number per 100 mL.	Plate count	p. 143 ³	D6503-99 ⁹ .	Enteroler-t ^{®12,22} .
	MPN ^{6,8} , multiple tube/multiple well, or. MF ^{2,5,6,7,8} two step, or 1106.1 ²³	9230 C-2007. 9230 C-2007.	D5259-92 ⁹ .	
	Single step, or	1600 ²⁴		
Protozoa:	Plate count	p. 143 ³ .			
8. <i>Cryptosporidium</i>	Filtration/IMS/FA	1622 ²⁵ , 1623 ²⁶ .			
9. <i>Giardia</i>	Filtration/IMS/FA	1623 ²⁶			

Table 1H notes:

¹ The method must be specified when results are reported.

² A 0.45-µm membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

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³ Microbiological Methods for Monitoring the Environment, Water, and Wastes. EPA/600/8-78/017. 1978. US EPA.

⁴ U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. USGS.

⁵ Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

⁶ Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

⁷ When the MF method has not been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

⁸ To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

⁹ Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. 2000, 1999, 1996. ASTM International.

¹⁰ Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. 1995. AOAC International.

¹¹ The multiple-tube fermentation test is used in 9221B.1–2006. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.

¹² These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β -glucuronidase produced by *E. coli*.

¹³ After prior enrichment in a presumptive medium for total coliform using 9221B.1–2006, all presumptive tubes or bottles showing any amount of gas, growth, or acidity within 48 h \pm 3 h of incubation shall be submitted to 9221F–2006. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 $\mu\text{g}/\text{mL}$ of MUG may be used.

¹⁴ Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple-well procedures, Quanti-Tray® or Quanti-Tray®/2000, and the MPN calculated from the table provided by the manufacturer.

¹⁵ Colilert-18® is an optimized formulation of the Colilert® for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 °C, rather than the 24 h required for the Colilert® test, and is recommended for marine water samples.

¹⁶ Descriptions of the Colilert®, Colilert-18®, Quanti-Tray®, and Quanti-Tray®/2000 may be obtained from IDEXX Laboratories Inc.

¹⁷ A description of the mColiBlue24® test may be obtained from Hach Company.

¹⁸ Subject total coliform positive samples determined by 9222B–1997 or other membrane filter procedure to 9222G–1997 using NA-MUG media.

¹⁹ Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC), EPA-821-R-10-002. March 2010. US EPA.

²⁰ Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC), EPA-821-R-09-007. December 2009. US EPA.

²¹ Preparation and use of MI agar with a standard membrane filter procedure is set forth in the article, Brenner et al. 1993. New Medium for the Simultaneous Detection of Total Coliform and *Escherichia coli* in Water. Appl. Environ. Microbiol. 59:3534–3544 and in Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium), EPA 821-R-02-024, September 2002, US EPA.

²² A description of the Enterolert® test may be obtained from IDEXX Laboratories Inc.

²³ Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esclulin Iron Agar (mE-EIA), EPA-821-R-09-015. December 2009. US EPA.

²⁴ Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl- β -D-Glucoside Agar (mEI), EPA-821-R-09-016. December 2009. US EPA.

²⁵ Method 1622 uses a filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of *Cryptosporidium*. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA, EPA-821-R-05-001. December 2005. US EPA.

²⁶ Method 1623 uses a filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts. Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. EPA-821-R-05-002. December 2005. US EPA.

(b) The documents required in this section are incorporated by reference into this section with approval of the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies of the documents may be obtained from the sources listed in paragraph (b) of this section. Documents may be inspected at EPA's Water Docket, EPA West, 1301 Constitution Avenue NW., Room B102, Washington, DC (Telephone: 202-566-2426); or at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030, or go to: http://www.archives.gov/federal_register/

code_of_federal_regulations/ibr_locations.html. These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the FEDERAL REGISTER. The full texts of the methods from the following references which are cited in Tables IA, IB, IC, ID, IE, IF, IG and IH are incorporated by reference into this regulation and may be obtained from the source identified. All costs cited are subject to change and must be verified from the indicated source.

(1) Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available at <http://>

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water.epa.gov/scitech/methods/cwa/index.cfm or from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161

(i) Microbiological Methods for Monitoring the Environment, Water, and Wastes. 1978. EPA/600/8-78/017, Pub. No. PB-290329/A.S.

(A) Part III Analytical Methodology, Section B Total Coliform Methods, page 108. Table IA, Note 3; Table IH, Note 3.

(B) Part III Analytical Methodology, Section B Total Coliform Methods, 2.6.2 Two-Step Enrichment Procedure, page 111. Table IA, Note 3; Table IH, Note 3.

(C) Part III Analytical Methodology, Section B Total Coliform Methods, 4 Most Probable Number (MPN) Method, page 114. Table IA, Note 3; Table IH, Note 3.

(D) Part III Analytical Methodology, Section C Fecal Coliform Methods, 2 Direct Membrane Filter (MF) Method, page 124. Table IA, Note 3; Table IH, Note 3.

(E) Part III, Analytical Methodology, Section C Fecal Coliform Methods, 5 Most Probable Number (MPN) Method, page 132. Table IA, Note 3; Table IH, Note 3.

(F) Part III Analytical Methodology, Section D Fecal Streptococci, 2 Membrane Filter (MF) Method, page 136. Table IA, Note 3; Table IH, Note 3.

(G) Part III Analytical Methodology, Section D Fecal Streptococci, 4 Most Probable Number Method, page 139. Table IA, Note 3; Table IH, Note 3.

(H) Part III Analytical Methodology, Section D Fecal Streptococci, 5 Pour Plate Method, page 143. Table IA, Note 3; Table IH, Note 3.

(ii) [Reserved]

(2) Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm>.

(i) Method 300.1 (including Errata Cover Sheet, April 27, 1999), Determination of Inorganic Ions in Drinking Water by Ion Chromatography, Revision 1.0, 1997. Table IB, Note 52.

(ii) Method 551, Determination of Chlorination Disinfection Byproducts and Chlorinated Solvents in Drinking Water by Liquid-Liquid Extraction and

Gas Chromatography With Electron-Capture Detection. 1990. Table IF.

(3) National Exposure Risk Laboratory-Cincinnati, U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available from <http://water.epa.gov/scitech/methods/cwa/index.cfm> or from the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Telephone: 800-553-6847.

(i) Methods for the Determination of Inorganic Substances in Environmental Samples. August 1993. EPA/600/R-93/100, Pub. No. PB 94120821. Table IB, Note 52.

(A) Method 180.1, Determination of Turbidity by Nephelometry. Revision 2.0. Table IB, Note 52.

(B) Method 300.0, Determination of Inorganic Anions by Ion Chromatography. Revision 2.1. Table IB, Note 52.

(C) Method 335.4, Determination of Total Cyanide by Semi-Automated Colorimetry. Revision 1.0. Table IB, Notes 52 and 57.

(D) Method 350.1, Determination of Ammonium Nitrogen by Semi-Automated Colorimetry. Revision 2.0. Table IB, Notes 30 and 52.

(E) Method 351.2, Determination of Total Kjeldahl Nitrogen by Semi-Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(F) Method 353.2, Determination of Nitrate-Nitrite Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(G) Method 365.1, Determination of Phosphorus by Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(H) Method 375.2, Determination of Sulfate by Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(I) Method 410.4, Determination of Chemical Oxygen Demand by Semi-Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(ii) Methods for the Determination of Metals in Environmental Samples, Supplement I. May 1994. EPA/600/R-94/111, Pub. No. PB 95125472. Table IB, Note 52.

(A) Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry. Revision 4.4. Table IB, Note 52.

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- (B) Method 200.8, Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma Mass Spectrometry. Revision 5.3. Table IB, Note 52.
- (C) Method 200.9, Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry. Revision 2.2. Table IB, Note 52.
- (D) Method 218.6, Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater, and Industrial Wastewater Effluents by Ion Chromatography. Revision 3.3. Table IB, Note 52.
- (E) Method 245.1, Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry. Revision 3.0. Table IB, Note 52.
- (4) National Exposure Risk Laboratory-Cincinnati, U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm>.
- (i) EPA Method 200.5, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry. Revision 4.2, October 2003. EPA/600/R-06/115. Table IB, Note 68.
- (ii) EPA Method 525.2, Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry. Revision 2.0, 1995. Table ID, Note 10.
- (5) Office of Research and Development, Cincinnati OH. U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm> or from ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati OH 45268.
- (i) Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater. 1978. Table IC, Note 3; Table ID, Note 3.
- (ii) Methods for Chemical Analysis of Water and Wastes. March 1979. EPA-600/4-79-020. Table IB, Note 1.
- (iii) Methods for Chemical Analysis of Water and Wastes. Revised March 1983. EPA-600/4-79-020. Table IB, Note 1.
- (A) Method 120.1, Conductance, Specific Conductance, μhos at 25 °C. Revision 1982. Table IB, Note 1.
- (B) Method 130.1, Hardness, Total (mg/L as CaCO_3), Colorimetric, Automated EDTA. Issued 1971. Table IB, Note 1.
- (C) Method 150.2, pH, Continuous Monitoring (Electrometric). December 1982. Table IB, Note 1.
- (D) Method 160.4, Residue, Volatile, Gravimetric, Ignition at 550 °C. Issued 1971. Table IB, Note 1.
- (E) Method 206.5, Arsenic, Sample Digestion Prior to Total Arsenic Analysis by Silver Diethyldithiocarbamate or Hydride Procedures. Issued 1978. Table IB, Note 1.
- (F) Method 231.2, Gold, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.
- (G) Method 245.2, Mercury, Automated Cold Vapor Technique. Issued 1974. Table IB, Note 1.
- (H) Method 252.2, Osmium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.
- (I) Method 253.2, Palladium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.
- (J) Method 255.2, Platinum, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.
- (K) Method 265.2, Rhodium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.
- (L) Method 279.2, Thallium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.
- (M) Method 283.2, Titanium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.
- (N) Method 289.2, Zinc, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.
- (O) Method 310.2, Alkalinity, Colorimetric, Automated, Methyl Orange. Revision 1974. Table IB, Note 1.
- (P) Method 351.1, Nitrogen, Kjeldahl, Total, Colorimetric, Automated Phenate. Revision 1978. Table IB, Note 1.
- (Q) Method 352.1, Nitrogen, Nitrate, Colorimetric, Brucine. Issued 1971. Table IB, Note 1.
- (R) Method 365.3, Phosphorus, All Forms, Colorimetric, Ascorbic Acid, Two Reagent. Issued 1978. Table IB, Note 1.
- (S) Method 365.4, Phosphorus, Total, Colorimetric, Automated, Block

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Digestor AA II. Issued 1974. Table IB, Note 1.

(T) Method 410.3, Chemical Oxygen Demand, Titrimetric, High Level for Saline Waters. Revision 1978. Table IB, Note 1.

(U) Method 420.1, Phenolics, Total Recoverable, Spectrophotometric, Manual 4-AAP With Distillation. Revision 1978. Table IB, Note 1.

(iv) Prescribed Procedures for Measurement of Radioactivity in Drinking Water. 1980. EPA-600/4-80-032. Table IE.

(A) Method 900.0, Gross Alpha and Gross Beta Radioactivity. Table IE.

(B) Method 903.0, Alpha-Emitting iRadio Isotopes. Table IE.

(C) Method 903.1, Radium-226, Radon Emanation Technique. Table IE.

(D) Appendix B, Error and Statistical Calculations. Table IE.

(6) Office of Science and Technology, U.S. Environmental Protection Agency, Washington DC (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm>.

(i) Method 1625C, Semivolatile Organic Compounds by Isotope Dilution GCMS. 1989. Table IF.

(ii) [Reserved]

(7) Office of Water, U.S. Environmental Protection Agency, Washington DC (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm> or from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

(i) Method 1631, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. Revision E, August 2002. EPA-821-R-02-019, Pub. No. PB2002-108220. Table IB, Note 43.

(ii) Kelada-01, Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate. Revision 1.2, August 2001. EPA 821-B-01-009, Pub. No. PB 2001-108275. Table IB, Note 55.

(iii) In the compendium *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*. July 1998. EPA 821-B-98-016, Pub. No. PB95201679. Table IF, Note 1.

(A) EPA Method 1666, Volatile Organic Compounds Specific to the Pharmaceutical Industry by Isotope Dilution GC/MS. Table IF, Note 1.

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(B) EPA Method 1667, Formaldehyde, Isobutyraldehyde, and Furfural by Derivatization Followed by High Performance Liquid Chromatography. Table IF.

(C) Method 1671, Volatile Organic Compounds Specific to the Pharmaceutical Manufacturing Industry by GC/FID. Table IF.

(iv) Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I. Revision I, August 1993. EPA 821-R-93-010A, Pub. No. PB 94121654. Tables ID, IG.

(A) Method 608.1, Organochlorine Pesticides. Table ID, Note 10; Table IG, Note 3.

(B) Method 608.2, Certain Organochlorine Pesticides. Table ID, Note 10; Table IG, Note 3.

(C) Method 614, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3.

(D) Method 614.1, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3.

(E) Method 615, Chlorinated Herbicides. Table ID, Note 10; Table IG, Note 3.

(F) Method 617, Organohalide Pesticides and PCBs. Table ID, Note 10; Table IG, Note 3.

(G) Method 619, Triazine Pesticides. Table ID, Note 10; Table IG, Note 3.

(H) Method 622, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3.

(I) Method 622.1, Thiophosphate Pesticides. Table ID, Note 10; Table IG, Note 3.

(J) Method 627, Dinitroaniline Pesticides. Table ID, Note 10; Table IG, Notes 1 and 3.

(K) Method 629, Cyanazine. Table IG, Note 3.

(L) Method 630, Dithiocarbamate Pesticides. Table IG, Note 3.

(M) Method 630.1, Dithiocarbamate Pesticides. Table IG, Note 3.

(N) Method 631, Benomyl and Carbendazim. Table IG, Note 3.

(O) Method 632, Carbamate and Urea Pesticides. Table ID, Note 10; Table IG, Note 3.

(P) Method 632.1, Carbamate and Amide Pesticides. Table IG, Note 3.

(Q) Method 633, Organonitrogen Pesticides. Table IG, Note 3.

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- (R) Method 633.1, Neutral Nitrogen-Containing Pesticides. Table IG, Note 3.
- (S) Method 637, MBTS and TCMTB. Table IG, Note 3.
- (T) Method 644, Picloram. Table IG, Note 3.
- (U) Method 645, Certain Amine Pesticides and Lethane. Table IG, Note 3.
- (V) Method 1656, Organohalide Pesticides. Table ID, Note 10; Table IG, Notes 1 and 3.
- (W) Method 1657, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3.
- (X) Method 1658, Phenoxy-Acid Herbicides. Table IG, Note 3.
- (Y) Method 1659, Dazomet. Table IG, Note 3.
- (Z) Method 1660, Pyrethrins and Pyrethroids. Table IG, Note 3.
- (AA) Method 1661, Bromoxynil. Table IG, Note 3.
- (BB) Ind-01. Methods EV-024 and EV-025, Analytical Procedures for Determining Total Tin and Triorganotin in Wastewater. Table IG, Note 3.
- (v) Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume II. August 1993. EPA 821-R-93-010B, Pub. No. PB 94166311. Table IG.
- (A) Method 200.9, Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry. Table IG, Note 3.
- (B) Method 505, Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products in Water by Microextraction and Gas Chromatography. Table ID, Note 10; Table IG, Note 3.
- (C) Method 507, The Determination of Nitrogen- and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector. Table ID, Note 10; Table IG, Note 3.
- (D) Method 508, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector. Table ID, Note 10; Table IG, Note 3.
- (E) Method 515.1, Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector. Table IG, Notes 2 and 3.
- (F) Method 515.2, Determination of Chlorinated Acids in Water Using Liquid-Solid Extraction and Gas Chromatography with an Electron Capture Detector. Table IG, Notes 2 and 3.
- (G) Method 525.1, Determination of Organic Compounds in Drinking Water by Liquids-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry. Table ID, Note 10; Table IG, Note 3.
- (H) Method 531.1, Measurement of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post-Column Derivatization. Table ID, Note 10; Table IG, Note 3.
- (I) Method 547, Determination of Glyphosate in Drinking Water by Direct-Aqueous-Injection HPLC, Post-Column Derivatization, and Fluorescence Detection. Table IG, Note 3.
- (J) Method 548, Determination of Endothall in Drinking Water by Aqueous Derivatization, Liquid-Solid Extraction, and Gas Chromatography with Electron-Capture Detector. Table IG, Note 3.
- (K) Method 548.1, Determination of Endothall in Drinking Water by Ion-Exchange Extraction, Acidic Methanol Methylation and Gas Chromatography/Mass Spectrometry. Table IG, Note 3.
- (L) Method 553, Determination of Benzidines and Nitrogen-Containing Pesticides in Water by Liquid-Liquid Extraction or Liquid-Solid Extraction and Reverse Phase High Performance Liquid Chromatography/Particle Beam/Mass Spectrometry Table ID, Note 10; Table IG, Note 3.
- (M) Method 555, Determination of Chlorinated Acids in Water by High Performance Liquid Chromatography With a Photodiode Array Ultraviolet Detector. Table IG, Note 3.
- (vi) In the compendium *Methods for the Determination of Organic Compounds in Drinking Water*. Revised July 1991, December 1998. EPA-600/4-88-039, Pub. No. PB92-207703. Table IF.
- (A) EPA Method 502.2, Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series. Table IF.
- (B) [Reserved]
- (vii) In the compendium *Methods for the Determination of Organic Compounds in Drinking Water-Supplement II*. August

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1992. EPA-600/R-92-129, Pub. No. PB92-207703. Table IF.

(A) EPA Method 524.2, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry. Table IF.

(B) [Reserved]

(viii) Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition. October 2002. EPA 821-R-02-012, Pub. No. PB2002-108488. Table IA, Note 26.

(ix) Short-Term Methods for Measuring the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, Fourth Edition. October 2002. EPA 821-R-02-013, Pub. No. PB2002-108489. Table IA, Note 27.

(x) Short-Term Methods for Measuring the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, Third Edition. October 2002. EPA 821-R-02-014, Pub. No. PB2002-108490. Table IA, Note 28.

(8) Office of Water, U.S. Environmental Protection Agency, Washington DC (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm>.

(i) Method 245.7, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry. Revision 2.0, February 2005. EPA-821-R-05-001. Table IB, Note 17.

(ii) Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). March 2010. EPA-621-R-10-002. Table IH, Note 19.

(iii) Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE-EIA). December 2009. EPA-621-R-09-015. Table IH, Note 23.

(iv) Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl- β -D-Glucoside Agar (mEI). December 2009. EPA-821-R-09-016. Table IA, Note 25; Table IH, Note 24.

(v) Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC). December 2009. EPA-821-R-09-007. Table IA, Note 22; Table IH, Note 20.

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(vi) Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium). September 2002. EPA-821-R-02-024. Table IH, Note 21.

(vii) Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. December 2005. EPA-821-R-05-001. Table IH, Note 25.

(viii) Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. December 2005. EPA-821-R-05-002. Table IH, Note 26.

(ix) Method 1627, Kinetic Test Method for the Prediction of Mine Drainage Quality. December 2011. EPA-821-R-09-002. Table IB, Note 69.

(x) Method 1664, n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. Revision A, February 1999. EPA-821-R-98-002. Table IB, Notes 38 and 42.

(xi) Method 1664, n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. Revision B, February 2010. EPA-821-R-10-001. Table IB, Notes 38 and 42.

(xii) Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels. July 1996. Table IB, Note 43.

(xiii) Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using Lauryl Tryptose Broth (LTB) and EC Medium. April 2010. EPA-821-R-10-003. Table IA, Note 15.

(xiv) Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A-1 Medium. July 2006. EPA 821-R-06-013. Table IA, Note 20.

(xv) Method 1682: *Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium. July 2006. EPA 821-R-06-014. Table IA, Note 23.

(9) American National Standards Institute, 1430 Broadway, New York NY 10018.

(i) ANSI. American National Standard on Photographic Processing

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Effluents. April 2, 1975. Table IB, Note 9.

(ii) [Reserved]

(10) American Public Health Association, 1015 15th Street NW., Washington, DC 20005. Standard Methods Online is available through the Standard Methods Web site (<http://www.standardmethods.org>).

(i) Standard Methods for the Examination of Water and Wastewater. 14th Edition, 1975. Table IB, Notes 17 and 27.

(ii) Standard Methods for the Examination of Water and Wastewater. 15th Edition, 1980, Table IB, Note 30; Table ID.

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- (viii) OFR 97-198, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1997. Table IB, Note 47.
- (ix) OFR 98-165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-Water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry. 1998. Table IB, Note 50.
- (x) OFR 98-639, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace—Atomic Absorption Spectrometry. 1999. Table IB, Note 49.
- (xi) OFR 00-170, Methods of Analysis by the U.S. Geological Survey National

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Water Quality Laboratory—Determination of Ammonium Plus Organic Nitrogen by a Kjeldahl Digestion Method and an Automated Photometric Finish that Includes Digest Cleanup by Gas Diffusion. 2000. Table IB, Note 45.

(xii) Water-Resources Investigation Report 01-4098, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Moderate-Use Pesticides and Selected Degradates in Water by C-18 Solid-Phase Extraction and Gas Chromatography/Mass Spectrometry. 2001. Table ID, Note 13.

(xiii) Water-Resources Investigations Report 01-4132, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Organic Plus Inorganic Mercury in Filtered and Unfiltered Natural Water With Cold Vapor-Atomic Fluorescence Spectrometry. 2001. Table IB, Note 71.

(xiv) Water-Resources Investigation Report 01-4134, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Pesticides in Water by Graphitized Carbon-Based Solid-Phase Extraction and High-Performance Liquid Chromatography/Mass Spectrometry. 2001. Table ID, Note 12.

(xv) Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, editors, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. Table IB, Note 8.

(xvi) Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1989. Table IB, Note 2.

(xvii) Methods for the Determination of Organic Substances in Water and Fluvial Sediments. Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3. 1987. Table IB, Note 24; Table ID, Note 4.

(xviii) Techniques and Methods Book 5-B1, Determination of Elements in Natural-Water, Biota, Sediment and Soil Samples Using Collision/Reaction Cell Inductively Coupled Plasma-Mass Spectrometry. Chapter 1, Section B, Methods of the National Water Quality

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Laboratory, Book 5, Laboratory Analysis. 2006. Table IB, Note 70.

(xix) U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. Table IA, Note 4; Table IH, Note 4.

(xx) Water Temperature—Influential Factors, Field Measurement and Data Presentation, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1. 1975. Table IB, Note 32.

(34) Waters Corporation, 34 Maple Street, Milford MA 01757. Telephone: 508-482-2131, Fax: 508-482-3625.

(i) Method D6508, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte. Revision 2, December 2000. Table IB, Note 54.

(ii) [Reserved]

(c) Under certain circumstances, the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director upon recommendation of the Alternate Test Procedure Program Coordinator, Washington, DC.

(d) Under certain circumstances, the Administrator may approve additional alternate test procedures for nationwide use, upon recommendation by the Alternate Test Procedure Program Coordinator, Washington, DC.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters are cited in Tables IA, IB, IC, ID, IE, IF, IG, and IH are prescribed in Table II. Information in the table takes precedence over information in specific methods or elsewhere. Any person may apply for a change from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for such limited use changes may be made by letters to the Regional Alternative

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Test Procedure (ATP) Program Coordinator or the permitting authority in the Region in which the discharge will occur. Sufficient data should be provided to assure such changes in sample preservation, containers or holding times do not adversely affect the integrity of the sample. The Regional ATP Coordinator or permitting authority will review the application and then notify the applicant and the appropriate State agency of approval or re-

jection of the use of the alternate test procedure. A decision to approve or deny any request on deviations from the prescribed Table II requirements will be made within 90 days of receipt of the application by the Regional Administrator. An analyst may not modify any sample preservation and/or holding time requirements of an approved method unless the requirements of this section are met.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and <i>E. coli</i> ...	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ^{22,23}
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
8. <i>Salmonella</i>	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
Table IA—Aquatic Toxicity Tests:			
9–12. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C ¹⁶	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days.
2. Alkalinity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days.
3. Ammonia	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
10. Boron	P, FP, or Quartz	HNO ₃ to pH <2	6 months.
11. Bromide	P, FP, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous.	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
16. Chloride	P, FP, G	None required	28 days.
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes.
21. Color	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
23–24. Cyanide, total or available (or CATC) and free.	P, FP, G	Cool, ≤6 °C ¹⁸ , NaOH to pH >10 ^{5,6} , reducing agent if oxidizer present.	14 days.
25. Fluoride	P	None required	28 days.
27. Hardness	P, FP, G	HNO ₃ or H ₂ SO ₄ to pH <2.	6 months.
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes.
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
Table IB—Metals: ⁷			
18. Chromium VI	P, FP, G	Cool, ≤6 °C ¹⁸ , pH = 9.3–9.7 ²⁰ .	28 days.
35. Mercury (CVAA)	P, FP, G	HNO ₃ to pH <2	28 days.
35. Mercury (CVAFS)	FP, G; and FP-lined cap ¹⁷ .	5 mL/L 12N HCl or 5 mL/L BrCl ¹⁷ .	90 days. ¹⁷
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO ₃ to pH <2, or at least 24 hours prior to analysis ¹⁹ .	6 months.
38. Nitrate	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
40. Nitrite	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
41. Oil and grease	G	Cool to ≤6 °C ¹⁸ , HCl or H ₂ SO ₄ to pH <2.	28 days.

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TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
42. Organic Carbon	P, FP, G	Cool to $\leq 6^{\circ}\text{C}$ ¹⁸ , HCl, H ₂ SO ₄ , or H ₃ PO ₄ to pH <2.	28 days.
44. Orthophosphate	P, FP, G	Cool, to $\leq 6^{\circ}\text{C}$ ^{18,24}	Filter within 15 minutes; Analyze within 48 hours.
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes.
47. Winkler	G, Bottle and top	Fix on site and store in dark.	8 hours.
48. Phenols	G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
49. Phosphorous (elemental)	G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	48 hours.
50. Phosphorous, total	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
53. Residue, total	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	7 days.
54. Residue, Filterable	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	7 days.
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	7 days.
56. Residue, Settleable	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	48 hours.
57. Residue, Volatile	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	7 days.
61. Silica	P or Quartz	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	28 days.
64. Specific conductance	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	28 days.
65. Sulfate	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	28 days.
66. Sulfide	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , add zinc acetate plus sodium hydroxide to pH >9.	7 days.
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes.
68. Surfactants	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	48 hours.
69. Temperature	P, FP, G	None required	Analyze.
73. Turbidity	P, FP, G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	48 hours.
Table IC—Organic Tests: ⁸			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, FP-lined septum	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ .	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons.	G, FP-lined septum	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ⁹ .	14 days. ⁹
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , 0.008% Na ₂ S ₂ O ₃ , pH to 4–5 ¹⁰ .	14 days. ¹⁰
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols ¹¹ .	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , 0.008% Na ₂ S ₂ O ₃ .	7 days until extraction, 40 days after extraction.
7, 38. Benzidines ^{11,12}	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction. ¹³
14, 17, 48, 50–52. Phthalate esters ¹¹ ..	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	7 days until extraction, 40 days after extraction.
82–84. Nitrosamines ^{11,14}	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction, 40 days after extraction.
88–94. PCBs ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	1 year until extraction, 1 year after extraction.
54, 55, 75, 79. Nitroaromatics and isophorone ¹¹ .	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction, 40 days after extraction.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons ¹¹ .	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction, 40 days after extraction.
15, 16, 21, 31, 87. Haloethers ¹¹	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction, 40 days after extraction.
29, 35–37, 63–65, 107. Chlorinated hydrocarbons ¹¹ .	G, FP-lined cap	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	7 days until extraction, 40 days after extraction.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs ¹¹ .	G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , pH <9.	1 year.
Aqueous Samples: Field and Lab Preservation.	G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	7 days.
Solids and Mixed-Phase Samples: Field Preservation.	G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	24 hours.
Tissue Samples: Field Preservation	G	Cool, $\leq 6^{\circ}\text{C}$ ¹⁸	

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TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation.	G	Freeze, ≤ −10 °C	1 year.
114–118. Alkylated phenols	G	Cool, <6 °C, H ₂ SO ₄ to pH <2.	28 days until extraction, 40 days after extraction.
119. Adsorbable Organic Halides (AOX)	G	Cool, <6 °C, 0.008% Na ₂ S ₂ O ₃ , HNO ₃ to pH <2.	Hold at least 3 days, but not more than 6 months.
120. Chlorinated Phenolics	Cool, <6 °C, 0.008% Na ₂ S ₂ O ₃ , H ₂ SO ₄ to pH <2.	30 days until acetylation, 30 days after acetylation.
Table ID—Pesticides Tests:			
1–70. Pesticides ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , pH 5–9– ₁₅ .	7 days until extraction, 40 days after extraction.
Table IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO ₃ to pH <2	6 months.
Table IH—Bacterial Tests:			
1. <i>E. coli</i>	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₅ ⁵ .	8 hours. ²²
2. Enterococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₅ ⁵ .	8 hours. ²²
Table IH—Protozoan Tests:			
8. <i>Cryptosporidium</i>	LDPE; field filtration	1–10 °C	96 hours. ²¹
9. <i>Giardia</i>	LDPE; field filtration	1–10 °C	96 hours. ²¹

¹ “P” is for polyethylene; “FP” is fluoropolymer (polytetrafluoroethylene (PTFE); Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; “G” is glass; “PA” is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); “LDPE” is low density polyethylene.

² Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sample; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤ 6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤ 6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664 Rev. A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

³ When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirement of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

⁴ Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under Sec. 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sample; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0–6 °C, with minimum head space.

⁵ ASTM D7365–09a specifies treatment options for samples containing oxidants (e.g., chlorine). Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (20th and 21st editions) addresses dechlorination procedures.

⁶ Sampling, preservation and mitigating interferences in water samples for analysis of cyanide are described in ASTM D7365–09a. There may be interferences that are not mitigated by the analytical test methods or D7365–09a. Any technique for removal or suppression of interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide through quality control measures described in the analytical test method. Any removal or suppression technique not described in D7365–09a or the analytical test method must be documented along with supporting data.

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⁷ For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

⁸ Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

⁹ If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

¹⁰ The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

¹¹ When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤ 6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the use of benzidine).

¹² If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzdine.

¹³ Extracts may be stored up to 30 days at < 0 °C.

¹⁴ For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

¹⁵ The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na₂S₂O₃.

¹⁶ Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation.

¹⁷ Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

¹⁸ Aqueous samples must be preserved at ≤ 6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of “≤ 6 °C” is used in place of the “4 °C” and “< 4 °C” sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤ 6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

¹⁹ An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

²⁰ To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

²¹ Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

²² Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.

²³ For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB-EC) or 1681 (A-1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

²⁴ The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (i.e., that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (i.e., within 15 minutes of collection).

[38 FR 28758, Oct. 16, 1973]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting § 136.3, see the List of CFR Sections Affected, which appears in the Finding Aids section of the printed volume and at www.fdsys.gov.

§ 136.4 Application for and approval of alternate test procedures for nationwide use.

(a) A written application for review of an alternate test procedure (alternate method) for nationwide use may be made by letter via email or by hard copy in triplicate to the National Alternate Test Procedure (ATP) Program Coordinator (National Coordinator),

Office of Science and Technology (4303T), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave. NW., Washington, DC 20460. Any application for an alternate test procedure (ATP) under this paragraph (a) shall:

(1) Provide the name and address of the responsible person or firm making the application.